G.C.E. (A/L) Chemistry

Practical Handbook

2018

Department of Science
Faculty of Science and Technology
National Institute of Education
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Message from the Director General

The primary objective of this rationalized new curriculum is to transform the student population into a human resource replete with the skills and competencies required for the world of work, through embarking upon a pattern of education which is more student centered and activity based.

Within this rationalization process, the concepts of vertical and horizontal integration have been employed in order to build up competencies of students, from the foundation level to higher levels, and to avoid repetition of subject content in various subjects respectively and furthermore, to develop a curriculum that is implementable and student friendly.

Practical work is part and parcel of the teaching and learning in science. Most practitioners would agree that good- quality practical work can engage students, help them to develop important skills, and to understand scientific concepts and the process of scientific investigation.

The new Practical Handbook has been introduced with the aim of providing the teachers with necessary guidance for planning practical activities, engaging students effectively in the teaching learning process and to promote students' practical skills in the discipline of chemistry.

I wish to make use of this opportunity to thank and express my appreciation to the members of the Council and the Academic Affairs Board of the NIE, the resource persons who contributed to compile this Practical Handbook and other parties for their dedication in this matter.

Dr. (Mrs.) T. A. R. J. Gunasekara
Director General
National Institute of Education

Message from the Director

This handbook has been developed to support you in advancing your students to fluency in science. We have worked with teachers, university lecturers and curriculum experts to produce this handbook. The activities presented in this Practical Handbook satisfy curriculum objectives in the areas of Chemistry. Pre-lab and post-lab questions have been given together with each practical schedule focusing on students' prior knowledge and applications of the particular activity respectively.

There are three interconnected, but separate, reasons for doing practical work in a course of study. They are:

1. To support and consolidate scientific concepts (knowledge and understanding).
   This is done by applying and developing what is known and understood of abstract ideas and models. Through practical work we are able to make sense of new information and observations, and provide insights into the development of scientific thinking.
2. To develop investigative skills.
   These transferable skills include devising and investigating testable questions, identifying and controlling variables, and analyzing, interpreting and evaluating data.
3. To build and master practical skills.
   Such skills include using specialist equipment to take measurements, handling and manipulating equipment with confidence and fluency, recognizing hazards and planning how to minimize risk.

By focusing on the aforesaid reasons for carrying out a particular practical work, I hope, teachers will help their students to understand the subject better, to develop the skills of a scientist and to master the manipulative skills required for further study or jobs in STEM (Science, Technology, Engineering, Mathematics) subjects

In order to make practical work effective, the laboratory should be made a place of learning by doing. Guideline should be laid down by the teacher about the laboratory rules such as the following-

- Work area must be cleared.
- Strict attention should be paid to own work.
- Reagent stoppers should not be left on counter tops.
- Wastage of water, gas, electricity should be strictly avoided.
- Directions should be read and followed very carefully.
- Teachers should allow the student's entry in lab in his/her presence.
- Only those experiments should be done which are recommended by the teacher-incharge.

I take this opportunity to thank university lecturers, teachers, and the other resource persons who contributed to make this effort a success. Finally, I wish practicing chemistry teachers all the success in their effort to empower our young generation to be successful as members of information-rich as well as technologically advanced societies.

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Contents

Introduction to Laboratory Safety

Preparing for laboratory work

- Read the experiment and answer prelab questions.
- Familiarize yourself with the hazards of the materials in the laboratory as well as appropriate safe handling, storage and emergency protocols.
- Read labels and material before moving, handling or opening chemicals.
- Never use chemicals from an unlabeled containers.
- Familiarize with the location and operation of safety equipment (including fire extinguishers, eye wash, shower and first aid) and emergency exits.

During laboratory work

- Eating and drinking are not permitted in laboratories.
- Contact lenses are not recommended to be worn in the chemistry laboratory class.
- Safety goggles are recommended in the laboratories. Open shoes, such as slippers should never be worn in the laboratory.
- Tie back or otherwise restrain long hair when working in the lab.
- Never pipette by mouth, use a pipette filler.
- Report accidents and dangerous incidents promptly (all chemical spills, glassware breakage and fires).

Waste disposal

- All broken or cracked glassware should be disposed into the marked containers in the laboratory. Never put the broken glass into the waste paper bin.(Use red colour bin)
- Do not put solid waste into the sink. Put them into the appropriate bins.
- Collect acids and bases separately into the containers provided and neutralize before discarding.
- Collect and recycle organic solvents using distillation for reuse whenever possible.
- Never dispose any chemical to school waste collection yard.

Cleaning up before leaving

Perform a safety check at the end of each experiment and before leaving the lab.
Make sure to;

- Turn off gas, water, electricity, and heating apparatus.
- Return unused materials, equipment and apparatus to their proper storage locations.
- Properly dispose all waste materials as instructed.
- Clean up your work area when finished.

Wash your hands thoroughly before leaving the laboratory.

- Leave behind protective clothing (lab aprons, gloves etc.) when leaving the laboratory.

Potential laboratory hazards

Chemicals can gain entry into the body by;

- Inhalation of gases, vapour and particulate material (e.g.: mists, dusts, smoke, fumes).
- Absorption of liquids, solids, gases and vapour through the skin.
- Ingestion of chemicals directly or indirectly via contaminated foods and beverages and contact between mouth and contaminated hands (e.g.: nai1 biting, smoking).
- Injection of chemicals through needles and other contaminated laboratory sharps.

Precautions for handling corrosive materials

- Wear appropriate skin and eye protection
- Use the weakest concentration possible
- Handle in a chemical fume hood
- Use secondary containers when transporting and storing corrosives
- Always dilute by adding acids to water
- Dilute and mix slowly



Poison materials **Corrosive** **Flammable** **Explosive** **Oxidizer** **Radioactive**

Figure 1. Hazardous labels

Essential components in a chemical label

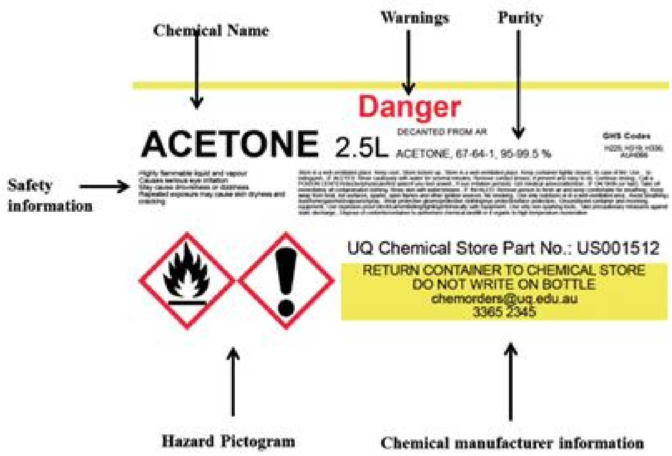
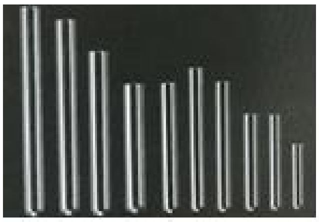


Figure 2. Components in a chemical label

Laboratory glass ware



Test tubes



Boiling tubes



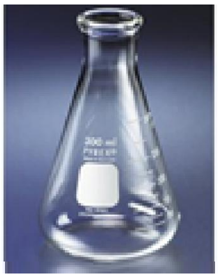
Beakers



Conical flasks



Volumetric flasks



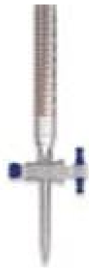
Titration flasks



Stoppered bottles



Measuring cylinders



Burette



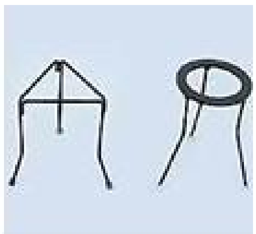
Pipettes



Round bottom flask



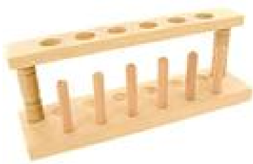
Funnel



Tripods



Pipette fillers



Test tube rack



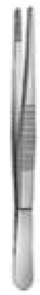
Watch glass



Test tube holders



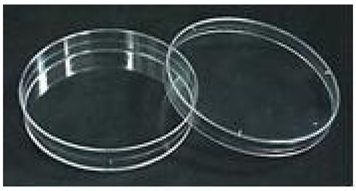
Pair of tongs



Forceps



Spatula



Petri dish



Mortar and pestle

Figure 3. Common glassware and items used in a chemistry lab

Basic Techniques in a Laboratory

Handling reagents

Reagent bottles should not be kept standing on the bench but must be placed on racks. Make sure you always keep the label facing you when placing bottles back in racks. When holding a bottle, always hold it with the side of the label.

Heating solution in a boiling tube

A Bunsen burner is used to heat solutions. It is important to know the principal parts in the flame in order to heat efficiently.



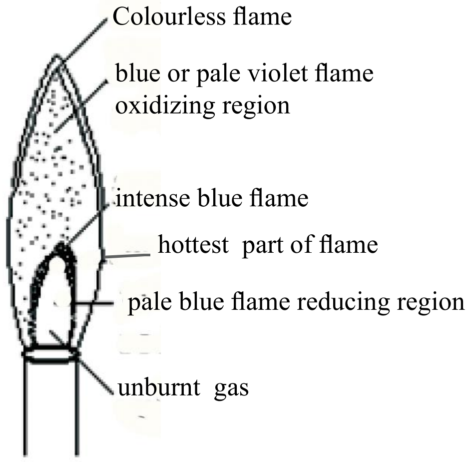


Figure 4. Major areas in a Bunsen flame

By adjusting the amount of air that is mixed with gas (using the air hole at the bottom of the burner body), different types of flames can be obtained for different purposes.

Type 1: Fully closed air hole - luminous (yellow)
Type 2: Air hole slightly open
Type 3: Partially opened air hole - inner blue cone with luminous tip, outer non-luminous zone
Type 4: Fully open-air hole - non luminous (blue)

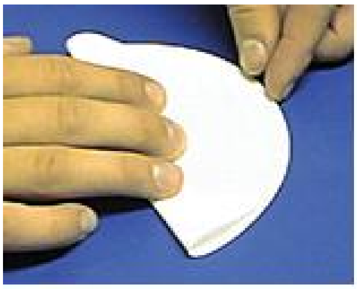
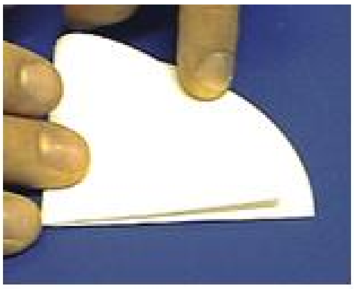
Type 3 is usually employed to heat solutions. The solution to be heated is poured into a boiling tube held with the test tube holder. The bottom part of the tube is heated in the yellow part of the flame while constantly moving the tube slowly in a circular path so that the solution is heated uniformly. Also, this will prevent cracking of the tube and overheating. The mouth of the boiling tube should be pointed away from the people

nearby. A small-medium size flame is sufficient for most types of heating and the height of the flame can be adjusted by controlling the gas flow.

Bunsen burners may not be available in all school laboratories. In that case demonstration of the above regions may have difficulty when using the normal burner.

Filtering

(1) Normal method: for the filtering of aqueous solutions



(2) Fluted filter paper method: for filtering of compounds in organic solvents

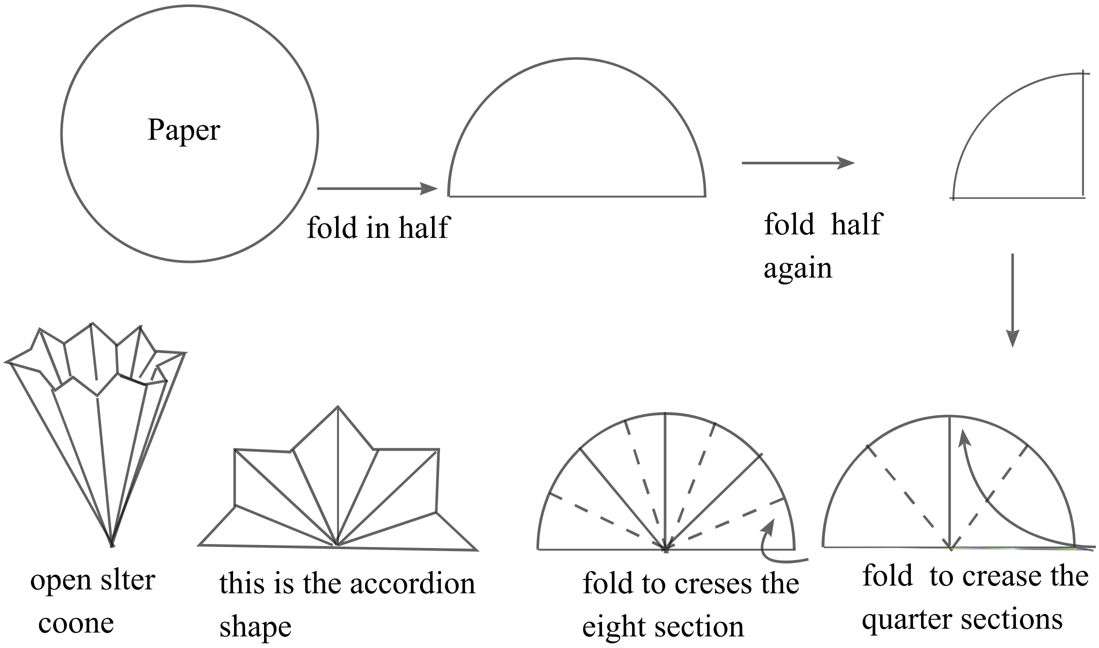


Figure 5. Fluted filter paper

A filter paper should be folded as indicated above depending on the type of mixture to be filtered. The filter paper should be well set against the walls of the funnel and moistened with distilled water. Care should be taken to prevent air trapping between the funnel and filter paper. The solution and the precipitate to be filtered are poured with the aid of a glass rod into the funnel. The height of the funnel should be adjusted such that there is a gap between the funnel and the test tube/beaker to which filtrate is collected. After pouring the solution, the test tube/beaker should be washed several times with distilled water and the washings should be added as described above. Finally the glass rod is washed with distilled water using the wash bottle in the funnel.

Gas handling

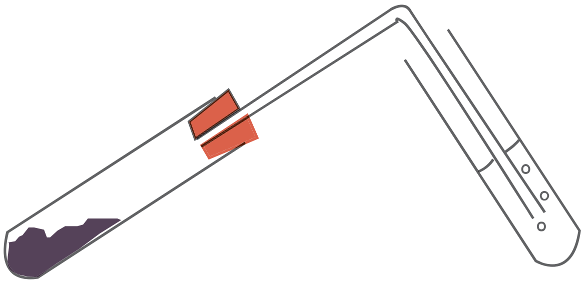


Figure 6. Apparatus for gas handling

Boiling tube contains the mixture which will emit gas. First it should be fixed to the holder and it is heated slowly until gas emission can be clearly seen from the tip of the gas delivery tube. Then test tube is placed in such a way that the gas is bubbled in to the solution. A sufficient volume of solution should be present in test tube so that the tip of the delivery tube is fully immersed in the solution. The whole set up should be swirled carefully to prevent over heating of the solution in boiling tube. The cork should well fit to the boiling tube in order to prevent leaking of gas. When heating is finished, first test tube is taken away from the delivery tube before boiling tube is taken away from the flame. This will prevent back flow of the solution in the test tube to boiling tube.

Evaporating solution to dryness



Figure 7. Evaporating dish

The solution is placed in an evaporating dish and the dish is placed on the clay triangle kept on the stand (ensure that the evaporating dish sits firmly on the clay triangle and there is no danger of tipping over). Heating is first done with a type 3 small flame to avoid splattering of solution in the evaporating dish. The size and temperature of the flame is gradually increased as the evaporation progresses if needed. In many cases, if the flame is removed while a small amount of liquid still remains in the evaporating dish, the heat already contained in the dish will be sufficient for the remaining liquid.

Use of burette

Burettes are used to measure accurately the volume of a liquid delivered, typically up to 0.05 cm^3 which is done by reading the meniscus before and after delivery. Most liquids including water, form a meniscus that is concave. Reading is taken by keeping the eye at the same level as the bottom of the meniscus of the liquid surface and estimating the level to the nearest 1/ 10th of a division, which correspond to 0.1 cm^3. A white background helps to get the reading more accurately.

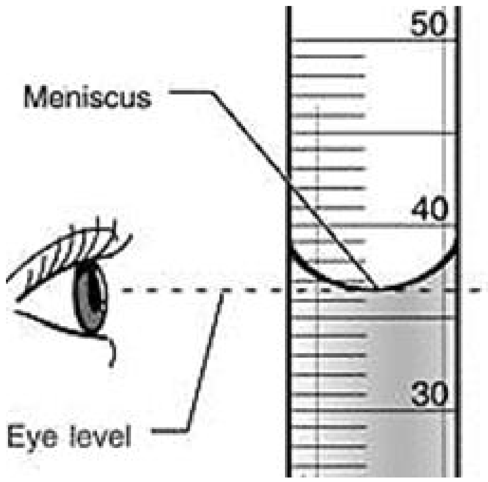


Figure 8. Proper method of reading a burette

Use of volumetric flask

Volumetric flasks are used to prepare solutions accurately. When preparing a solution, first, sample should be transferred to the flask using a funnel and funnel should be washed carefully and all the washings are also added to the flask and little amount of distilled water is added. Then the flask is stoppered well and shaken carefully to ensure complete dissolving of all the solute. Then distilled water is added up to the mark and further mixing is done to homogenize the solution. Care should be taken to handle the volumetric flask only by the neck to ensure no thermal expansion due heat in hands.

Use of balances





Figure 9. Electronic balance and four beam balance

The chemical balances are very sensitive equipment and require careful handling. The location of the balance must not be changed after setting the alignments. When transporting, must never press the pan area even by mistake by hand as the sensor might get damaged. Before taking a measurement always check whether the balance has proper alignments. When measuring a sample, make sure the total weight does not exceed the maximum weight limit of the balance. Chemicals should not be in direct contact with the top pan and do not forget to clean the balance immediately after use.

Post lab activities:

(i) Develop a floor plan to indicate location of safety equipment.

(ii) Discuss how you could reduce the use of chemicals in the laboratory and proper ways of disposing chemicals.

Experiment 1: Demonstrating the properties of cathode rays

Aims:

1. To provide necessary skills and knowledge to identify components in an electrical circuit used to operate a cathode ray tube
3. To observe and explain properties of cathode rays

Pre lab questions:

1. Name the sub atomic particles.
2. List the scientists who pioneered the discovery of sub atomic particles.
3. Who discovered the e/m ratio of cathode rays?

Introduction:

A special type of tubes called "Crookes tubes" is used to observe properties of cathode rays. Inside of these tubes contain a gas at low pressure. When observing electrical discharge, a significant voltage difference should be applied to two metal plates at both ends of the tube. Then the negatively charged plate called "cathode" emits invisible rays which pass to the other end of the tube. This end is specially coated with fluorescent material which glows when this invisible cathode rays fall on it. Figure 1.1 illustrates different types of Crookes tubes.

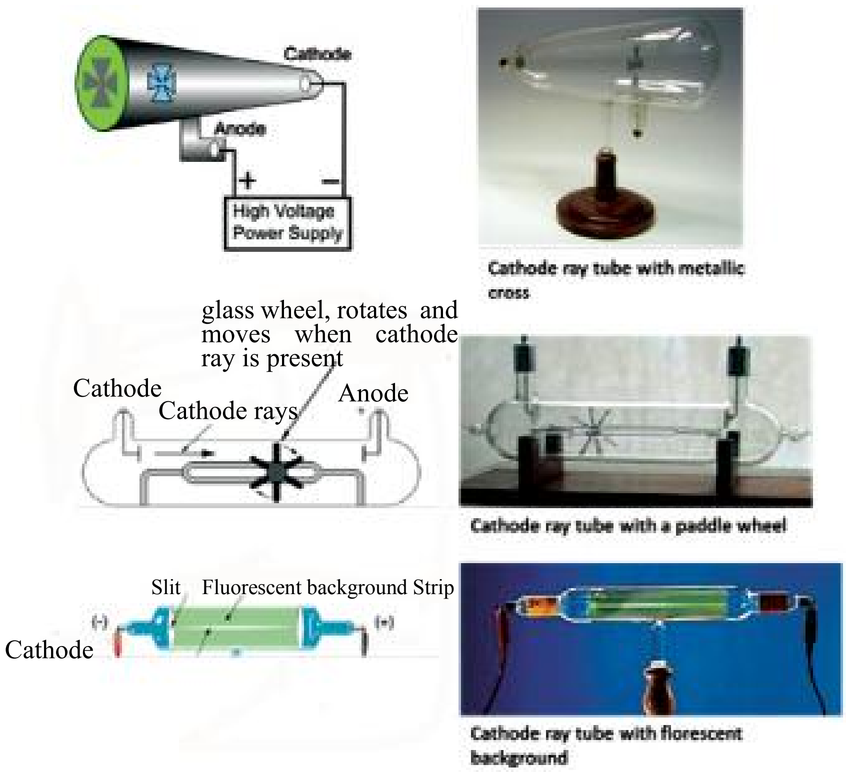


Figure 1.1 Different types of cathode ray tubes

Equipment required:

Equipment :
Cathode ray tubes (Crookes tubes)
Induction coil
6 V direct current source (Lead accumulator or Ni/Fe cell or power pack/ electronic high voltage supply
Connecting wires
Switch

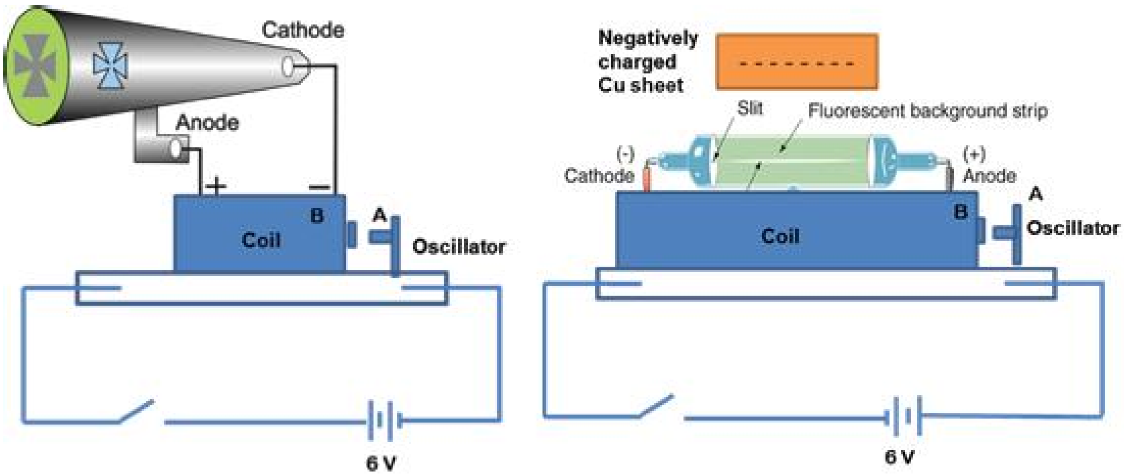


Figure 1.2. (a) Set-up for experiments I and II (b) Set-up for experiments III and IV

Precautions:

- Care should be taken to avoid electrical shock by keeping high voltage equipment or connecting wires away from human body contact.
- Make sure to keep the switch off to prevent current flowing through the circuit when not observing properties of cathode rays.

Procedure:

Experiment I:

- Set-up the experiment as illustrated in Figure 1.2(a)
- Close the switch to allow current flow through the circuit.
- Turn the oscillator screw left or right to create electrical spark between A and B.
- At this point green colour glow will appear inside the cathode ray tube. (if a green colour glow does not appear, change + and – ends of the voltage source.) This glow appears due to cathode rays.
- Open the switch to stop the current flow.
- Adjust the metallic cross inside the cathode ray tube vertically and allow the current to pass through the circuit again.

- Record your observations .
- After observations open the switch to stop the current flow through the circuit.

Experiment II

- Switch on the cathode ray tube with paddle wheels in the above circuit. Use the experimental set up in Figure 1.2.(a)
- Make sure to keep the tube horizontal much as possible.
- Close the switch to let current flow through the circuit. Record the observations.
- Now change the + and – terminals of the cells to make the current flow the in the opposite direction and observe any changes in the cathode ray tube.
- Stop the current flow at the end of the experiment.

Experiment III

- Arrange the experimental set-up as shown in Figure 1.2.(b).
- Arranged the circuit in a way that the Cu sheet is negatively charged.
- Then use this negatively charged Cu sheet to observe the behaviour of cathode rays in the presence of an electric field.
- Record your observations.
- Also record observations when the Cu sheet is moved below the cathode ray tube.

Experiment IV

- Use the experimental setup as in Figure 1.2.(b) with the cathode ray tube used in experiment III.
- Allow current to pass through the circuit and record observations.
- Next, hold a south pole of a magnet close to cathode rays. Record observations.
- Hold the north pole of a magnet close to cathode rays. Record observations.

Results and Discussion:
Experiment I:

Observations: ....
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What property of cathode rays is demonstrated by this experiment?

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Experiment II:

Observations: ………………………………………………………………………………
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What property of cathode rays is demonstrated by this experiment?

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Experiment III:

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What property of cathode rays is demonstrated by this experiment?

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Experiment IV:

Observations::

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What property of cathode rays is demonstrated by this experiment?

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Post lab questions:

1. Do you expect cathode rays to have a colour? Then explain how cathode rays are visualized as a greenish glow?
2. Why do you need to use a high voltage source?
3. List properties of cathode rays observed in this experiment.

Experiment 2: Displaying the shapes of molecules and ions using models

Aim:

To provide understanding on different types of bonding between atoms and orientation of atoms in 3D space for simple molecules and ions

Pre lab questions:

1. Briefly explain the difference among covalent bonding, ionic bonding and metallic bonding.
2. Write VSEPR theory and deduce the structure of molecules and ions giving suitable examples.
3. Write the maximum number of electrons allowed in the valence shell of each of the following atoms when making covalent bonding (Li, Be, B, C, N, O, F, Mg, Al, Si, P , S, Cl)

Introduction:

The type of bonding between atoms in a given molecule or ion is responsible for many physical characteristics such as melting point, boiling point, polarity, solubility, etc. Therefore, understanding the structure of the molecules and orientation of atoms in 3D space are important when explaining these physical properties. The shapes of molecules/ions can be predicted using VSEPR theory.

Equipment required:

Equipment
Molecular model kit
Alternative materials that can be used as models (sticks, lime, clay, balloons)
Pre-assembled model kits to show s and p orbitals



Figure 2.1. Molecular model kits

Procedure:

- Select the appropriate ball for the central atom looking at the number of bonds it can make.
- Build molecules listed below and deduce the shape.

Results

Molecule/ ion	Total number of electron pairs in valence shell around the central atom	Number of VSEPR pairs around the central atom	Number of lone electron pairs on the central atom	Electron pair geometry	Shape of the molecule/ ion
O_3					
$BeCl_2$					
BF_3					
CH_4					
NH_3					
H_2O					
PCl_3					
SF_6					
SF_4					
ClF_3					
XeF_2					
XeF_4					
IF_5					
NH_4^+					
SO_4^{2-}					
CO_3^{2-}					
NO_3^-					
O_2					
N_2					
H_2					

Consider one atom as a central atom for H_2 , N_2 and O_2

Discussion:

a. Discuss differences between model structure and the real structure of molecules to explain that models cannot be used 100% to represent a molecule. (Resonance structures)

Post lab questions:

1. Using models of H_2O and CH_4, explain how to determine if a molecule is polar or nonpolar.
2. Is CO_2 a polar or a nonpolar molecule? Give reasons.

Experiment 3: Handling glassware and four beam balance

Aims:

To provide necessary knowledge for students to

1. identify different types of glassware used in the laboratory and their purposes.
2. use four beam balance for weight measurements.

Pre lab question:

Read the introduction section in the laboratory manual and list different types of glassware you would expect to see in a chemistry laboratroy.

Introduction:

Glassware is an essential item that is available in almost all science laboratories. Therefore, proper handling and cleaning of glassware is necessary for the long term use. Glassware is different based on the purpose they are used for. In order to minimize errors during experiment, it is important to use appropriate glassware.

Broken glassware can cause injuries during laboratory classes. Therefore, it is important to inspect for cracks and breakages before using them in an experiment. A common mistake other than breakage is that glassware may crack if exposed to a sudden temperature difference (eg. accidentlly keeping heated glassware on a wet surface). Also, make sure to use clean glassware and items in the laboratory to ensure accurate results. The proper procedure to clean glassware is washing them first with a suitable liquid detergent followed by soaking glassware in a base bath (KOH/ ethanol for a brief period to remove any trace metals present). After rinsing glassware with the base bath, it should be dipped in an acid bath (dil. HCl) for about 30 minutes. Finally glassware is rinsed with water, distilled water and acetone prior to placing them in the drying cabinet. It is important not to place any volumetric glassware in an oven or a glassware drying cabinet to preserve calibration. Certain experimental set ups involving quick fit may require application of slight amount of grease to prevent breakages when dismantling at the end of experiments. At the end of each experiment it important to clean glassware the earliest possible. Also, read the introduction of the practical handbook for safe disposal of chemicals after use.



Figure 3.1. Four beam balance

There are different kinds of balances used in science laboratories for weight measurements. These could vary from simple four beam balance to a more sophisticated digital balance.

Equipment required:

Equipment
Glassware items listed in the table
Four/three beam balance
Weighing boats
Weight set (5g, 10g, 50g)

Precaution: Inspect glassware for breakages and contamination before use

Procedure:

3.1. Handling glassware:

- Identify following glassware using the introduction section of the laboratory manual and draw them in the following table.
- Discuss the use of each glassware and list any precautions when used in experiments.

3.2. Handling a four beam balance

- Identify different components in a four beam balance.
- Move all four sliders (movable masses) in the balance so that they read ‘zero’.
- Make sure that there is nothing on the pan and that it is clean.
- Check to see if the balance reads zero. If balance isn’t reading zero, turn the thumbscrew (zero adjust screw) to adjust the balance until it reads zero.
- Move the sliders, beginning with the largest of moving a slider causes the balance to tip, move the slider back to the previous position.
- Move the next slider until it tips the balance. Place it in the previous position.
- Move the final slider until the balance reads zero.
- Read each of the sliders and add their weights together.
- Practice making measurements using known weights given.

Results: 3.1 Experiment

Glassware / Item	Sketch	Purpose	Precautions
Test tubes			
Boiling tube			
Beaker			
Conical flask			
Titration flask			
Volumetric flask			
Round bottom flask			
Flat bottom flask			

Glassware / Item	Sketch	Purpose	Precautions
Stoppered bottle (reagent bottles)			
Wash bottle			
Funnel			
Measuring cylinder			
Pipette			
Pipette filler			
Burette			
Droppers			
Petri dish			
Watch glass			
Liebig condenser			
Crucible with lid			
Spatula			
Pair of tongs			
Forceps			
Test tube rack			
Test tube holders			

Handling a four beam balance

Object	Slider 1 reading (w_1) /g	Slider 2 reading (w_2)/g	Slider 3 reading (w_3) /g	Slider 4 reading (w_3) /g	Weight reading (w_1+ w_2+w_3+ w_4)/g

Discussion:

- Discuss how to categorize laboratory waste.
- Discuss the importance of maintaining MSDS sheets
- Discuss how to develop a method for safe disposal of laboratory waste.

Post lab questions:

1. List glassware that can be used for volume measurements? What is the minimum volume each glassware can measure?
2. Explain what steps you can take to remove glassware (test tubes, pipettes, burets) with contaminations.

Experiment 4: Preparation of standard solutions

Aims:

1. To gain the skill of using glassware and the four beam balance
2. To develop the skill of measuring volumes
3. To gain the competency to prepare solutions of required concentrations

Pre lab questions:

1. What are the methods of determining the composition of a solution?
2. Define the molar concentration.
3. What are the glassware used to prepare a solution of known concentration.

Introduction:

Concentration can be defined as follows.

$$\text{Concentration} = \frac{\text{Amount of the solute}}{\text{Volume of the solution}}$$

In a 1 mol dm^{-3} solution, 1 mol of solute is present in 1 dm^3 of the solution. According to that in a c mol dm^{-3} solution, c mol of solute is present in 1 dm^3 of the solution. If we know the amount of moles in a volume that we need to prepare, we can prepare the required volume of the solution needed. For example, assume that we want to prepare 250 cm^3 of 1 mol dm^{-3} Na_2CO_3 solution.

$$\text{Amount of } Na_2CO_3 \text{ present in that volume of the solution} = \frac{1 \text{ mol dm}^{-3} \times 250 \text{ cm}^3}{1000 \text{ cm}^3 \text{ dm}^{-3}}$$

$$= 0.25 \text{ mol}$$

$$\text{Mass of the 0.25 mol of } Na_2CO_3 = 106 \text{ g mol}^{-1} \times 0.25 \text{ mol}$$

$$= 26.5 \text{ g}$$

When this mass is dissolved in water and diluted up to 250 cm^3, the required solution is obtained.

Equipment and chemicals required:

Equipment	Chemicals
Volumetric flask (250 cm^3)	Na_2CO_3(s)
Beakers	
Funnel	
Watch glasses	
Four beam balance	
Wash bottle	

Precautions: Avoid touching the chemicals. The glassware should be well cleaned.

Procedure:

- Preparation of 250 cm^3 of 1 mol dm^{-3} Na_2CO_3 solution
- Keep the watch glass on the plate of the four beam balance and weigh it. Assume that it is w g.
- Add the required mass of Na_2CO_3 sample that should be measured and w g. (26.5+ w) g.
- Adjust the sliders of the beams indicating the above mass. Then the tip of the balance is below zero.
- Add solid Na_2CO_3 to the watch glass until the balance reads zero. Then the watch glass contains 26.5 g of solid Na_2CO_3
- Keep the funnel on the 250 cm^3 volumetric flask and add the Na_2CO_3 sample. Wash the watch glass with distilled water using the wash bottle. Wash the funnel with distilled water. Then remove the funnel and put the stopper and shake the volumetric flask until Na_2CO_3 is dissolved and add distilled water carefully up to the 250 cm^3 mark. Use the droper or a glass tube to adjust the final volume.
- Label the solution. On the label indicate the name of the solution, the concentration of the solution and the date that the solution was prepared.

Post lab questions:

1. Briefly explain how the following solutions are prepared.
   a. 500 cm^3 of 2 mol dm^{-3} NaOH solution. (Na = 23, O = 16, H = 1)
   b. 100 cm^3 of 0.2 mol dm^{-3} $K_2C_2O_4$ solution. (K = 39, C = 12, O = 16)
2. A student is saying that the concentration of the solution mentioned in (i) a) could be changed by a little amount. Discuss why a standard solution with an accurate concentration could not be prepared by NaOH.
3. Name primary standard substances that could be used to prepare solutions with accurate concentrations.

Experiment 5: **Experimental determination of molar volume of a gas.**

Aims:

1. To develop skills required to collect and measure gas volumes in the laboratory.
2. To calculate volume of one mole of oxygen gas under laboratory conditions.

Pre lab questions:

1. List reactions that liberate oxygen under laboratory conditions.
2. Discuss which of the above reaction is most suitable to collect and measure the volume of oxygen gas under lab conditions while stating why other reactions are not suitable.
3. What is the molar volume of a gas at standard temperature and pressure?

Introduction:

Standard molar volume of a gas is the volume of one mole of a gas at 0 °C and 1atm pressure (i.e standard temperature and pressure (STP)). Molar volume of a gas varies with temperature and pressure. In this experiment molar volume of oxygen gas is determined. Oxygen gas can be produced for the experiment using the following reaction.

$$2KMnO_4\,(s) \rightarrow K_2MnO_4\,(s) + MnO_2\,(s) + O_2\,(g)$$

In this reaction the only gaseous product is oxygen. Therefore, we can use weight loss during reaction to find the mass of oxygen produced and a water displacement method to find the volume of oxygen gas.

The pressure of the dry oxygen gas (P_{O_2}) is calculated using Dalton's Law of Partial Pressures:

$$P_{total} = P_{O_2} + P_{H_2O} \text{ or } P_{O_2} = P_{total} - P_{H_2O}$$

where P_{total} is the atmospheric pressure, and P_{H_2O} is the vapour pressure of water which is given in the table below.

Temperature / °C	Vapour pressure of water/ torr	Temperature /°C	Vapour pressure of water /torr
20	17.5	29	30.0
21	18.7	30	31.8
22	19.8	40	55.3
23	21.1	50	92.5
24	22.4	60	149.4
25	23.8	70	233.7
26	25.2	80	355.1
27	26.7	90	525.8
28	28.3	100	760.0

Then we can use combined gas law to find the volume of oxygen gas at STP.

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$$

Where P_1, V_1, T_1 are STP conditions and P_2 (Partial pressure of oxygen gas, P_{O_2}), V_2 (volume of oxygen gas measured) and T_2 room temperature) are laboratory conditions. Once V_1 at STP conditions is calculated, molar volume of oxygen can be calculated based on moles of oxygen gas produced.

Equipment and chemicals required:

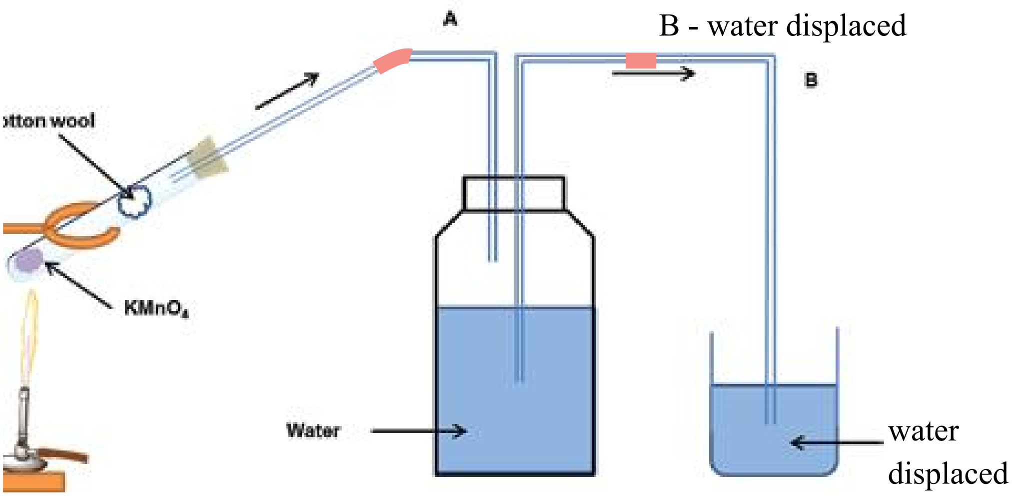
$$\text{Molar volume of oxygen gas} = \frac{\text{Volume of oxygen gas at STP (L)}}{\text{amount of moles of oxygen (mol)}}$$

Equipment	
Measuring cylinder (1L)	Thermometer
1L beaker	Barometer
Boiling tube with cork	Top pan balance
Delivery tube	Cotton wool
Glass bottle with cork	**Chemicals**
Rubber tubing	potassium permanganate ($KMnO_4$)
Boiling tube clamps	
Stands	
bunsn burner	

Experimental set-up

Oxygen gas is collected by displacement of water in the following experimental set-up.

Set-up I



Set-up II

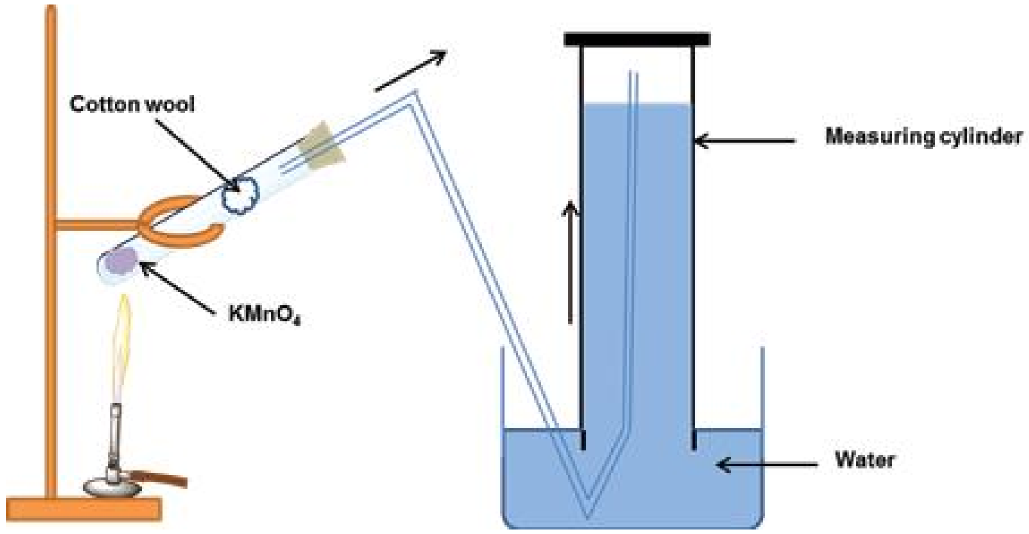


Figure 5.1. Experimental set-up

Precautions: Wear goggles. Make sure to use dry $KMnO_4$.

Procedure:

- Weigh about 5 g of dry $KMnO_4$ and transfer it into the dry boiling tube.
- Insert a cotton wool plug closer to the top of the boiling tube.
- Measure the total weight of the boiling tube, $KMnO_4$ and cotton wool (m_1).
- Set up the apparatus as shown in the above figure and gently heat the boiling tube.
- Continue heating until amount of water displaced due to oxygen gas is about 350- 400 cm^3. then allow the boiling tube to cool to room temperature.
- Measure the total weight of boiling tube and it's content again (m_2).
- Remove the rubber tube on side B when the water levels of the two containers are the same. Make sure to let remaining water in tube B is transferred to the beaker.
- Measure the volume of water displaced using the measuring cylinder.
- Record the room temperature and pressure.

Results :

	Readings
Weight of the boiling tube and it's content before heating (m_1)/g	
Weight of the boiling tube and it's content after heating (m_2)/g	
Volume of water displaced (i.e. Volume of oxygen gas)/ml	
Temperature /^oC	
Pressure/ mmHg	

Calculations:

a) Calculate pressure of dry O_2 (P_{O_2}) by subtracting the vapour pressure of water from the total pressure, (if set-up II is used)

$$P_{total} = P_{O_2} + P_{H_2O}$$

$$P_{O_2} = P_{total} - P_{H_2O}$$

where P_{total} is atmospheric pressure, and P_{H_2O} is saturated vapour pressure of water vapour at room temperature. Use the table provided to find the vapour pressure of water as a function of temperature.

P_{O_2} = ______________________ mm Hg

b) Convert this pressure from mm Hg to Nm^{-2} (760.0 mm Hg = 1.01×10^5 Nm^{-2})

P_{O_2} = ______________________ Nm^{-2}

c) Use the combined gas equation to calculate the volume of oxygen gas at STP.

Volume of O_2 at STP = ______________________ L

d) Amount of moles of oxygen gas produced

moles of oxygen gas produced during the experiment = $(m_1 - m_2)$ / 32 g mol^{-1}

$$\text{molar volume of oxygen gas} = \frac{\text{Volume of oxygen gas at STP (L)}}{\text{moles of oxygen (mol)}} = \text{______ L mol}^{-1}$$

e) Molar volume of oxygen gas

Post lab questions:

1. Discuss
   a. why cotton wool is inserted into the boiling tube.
   b. why it is important to let the boiling tube cool to room temperature before weighing the contents.
   c. why it is important to level water prior to removing the tube B.

2. Calculate the error percentage in comparison to the book value.

Experiment 6: Experimental determination of molar mass of magnesium using molar volume of hydrogen gas

Aim:

To develop the skill of the determination of relative atomic mass of metals.

Pre lab questions:

1. List different gasses that are not reacting with water.
2. List metals that react fast with dilute acids and write balanced chemical equations.
3. State ideal gas law and Dalton law of partial pressures. eqations, Define all terms.
4. List different techniques that can be used to collect gasses in laboratories.

Introduction:

For chemical reactions involving gases, gas volume measurements provide a convenient means of determining stoichiometric relationships. Hydrogen gas is evolved when certain metals are reacting with acids. In this experiment, amount of hydrogen gas produced in reaction of magnesium with dil. HCl acid is used to calculate the relative atomic mass of magnesium.

$$Mg\ (s) + 2HCl\ (aq) \rightarrow MgCl_2\ (aq) + H_2\ (g)$$

Hydrogen gas evolved in this reaction is collected in a long, thin graduated glass tube, using an eudiometer or using a burette, by displacement of a liquid, usually water. When magnesium reacts with the acid, the evolved hydrogen gas is collected by displacement of water and its volume is measured. The temperature of the gas is taken to be the same as the temperature of the water it is in contact with because, given a sufficient amount of time, the two will reach thermal equilibrium. The level of water in the eudiometer is adjusted so that it is equal to the level of water outside the eudiometer. This ensures that the pressure in the eudiometer is equal to the prevailing atmospheric pressure. Because hydrogen gas was collected above water, and water has a significant vapour pressure, to get the pressure of pure hydrogen (dry hydrogen), we must subtract the vapour pressure of water.

The pressure of the dry hydrogen gas (P_{H_2}) is calculated from Dalton law of partial \ pressures:

$$P_{total} = P_{H_2} + P_{H_2O} \quad so \quad P_{H_2} = P_{total} - P_{H_2O}$$

where P_{total} (the pressure in the eudiometer) is the atmospheric pressure, and P_{H_2O} (the water vapour pressure of water) is the pressure exerted by water vapour that has evaporated into the eudiometer. We will get the vapour pressure of water from the table below of vapour pressure vs. temperature.

Temperature/ ^{0}C	Vapour pressure of water/ torr	Temperature/ ^{0}C	Vapour pressure of water /torr
20	17.5	29	30.0
21	18.7	30	31.8
22	19.8	40	55.3
23	21.1	50	92.5
24	22.4	60	149.4
25	23.8	70	233.7
26	25.2	80	355.1
27	26.7	90	525.8
28	28.3	100	760.0

Since volume (*V*) of hydrogen gas is known, the number of moles of hydrogen gas collected (*n*) can then be calculated from the ideal gas law:

$$n = \frac{PV}{RT} \quad \text{(Use } P_{H_2} \text{ here, not } P_{total} \text{ and } R = 8.314\ \text{J K}^{-1}\ \text{mol}^{-1}\text{)}$$

Since stoichiometry between Mg and H_2 gas is 1:1, we can calculate the amount of Mg in moles reacted with HCl. The mass of magnesium strip that reacted is known, therefore, we can calculate the relative atomic mass of magnesium as follows.

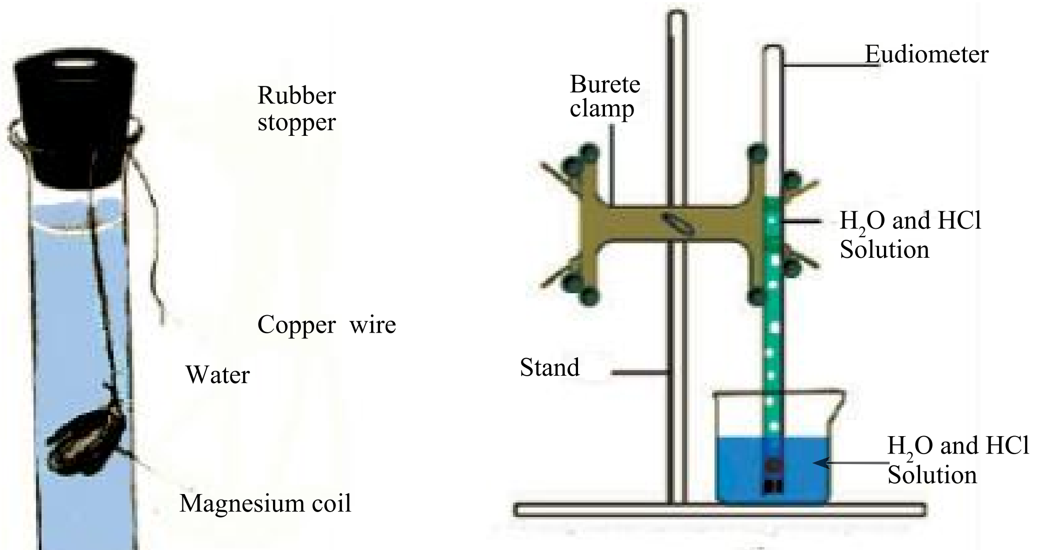
$$\text{molar mass of Mg} = \frac{\text{mass of Mg strip (g)}}{\text{amount of moles of Mg (mol)}}$$

Equipment and chemicals required:

Equipment	Chemicals
Eudiometer/burette	Magnesium strips
1L beaker	A piece of Cu wire
Burette clamps	2 M HCl
Burette stands	
Thermometer	
Barometer	

Experimental set-up

Hydrogen gas is collected by displacement of water in the following experimental set- up. A burette in upside down position can also be used in place of an eudiometer.



Inverted eduiometer illustrating gas collection and water displecement

Figure 6.1. Experimental set-up

Precautions: Wear gloves when handling acids.

Procedure:

- pour 5 cm^3 of 2 mol dm^{-3} HCl solution into the bottom of a 10 cm^3 graduated cylinder.
- Pour water gently on top of HCl carefully without mixing and add water so that the cylinder is completely filled with the liquid.
- Fix a 1.0 cm strip of magnesium (~ 0.05g) to the tapered end of a cork with a hole in it and place this cork on top of the graduated cylinder.
- Invert The cylinder was quickly and place the end under water in a 1L beaker filled with water.
- The reaction starts with the diffusion of HCl to the metal and the gas is captured in the inverted cylinder. When the reaction ceases, the levels of water in the cylinder and beaker are equalized and the volume of the gas is measured. The experiment is repeated three times.

Results:

	Trial 01	Trial 02	Trial 03	Average
Weight of Mg /g				
Volume of H_2 gas produced /cm^3				
Temperature / ^0C				
Partial pressure of H_2O/ torr				

Calculations:

a) Calculate pressure of dry H_2 (P_{H_2}) by subtracting the vapour pressure of water from the total pressure.

$$P_{total} = P_{H_2} + P_{H_2O}$$

$$P_{H_2} = P_{total} - P_{H_2O}$$

where P_{total} (the pressure in the eudiometer) is atmospheric pressure, and P_{H_2O} (the water vapour pressure of water) is the pressure exerted by water vapor that has evaporated into the eudiometer. Use the table provided on the last page to find the vapour pressure of water as a function of temperature.

P_{H_2} = ________________ mmHg

b) Convert this pressure from mm Hg to Nm^{-2} (760.0 Hg mm= 1.01 x 105 Nm^{-2})

P_{H_2} = ________________________ Nm^{-2}

c) Use the Ideal Gas Equation to calculate the amount of moles (n) of H_2 that you produced in your experiment (experimental yield). Make sure to use the correct units so that they match the units in the gas constant (R).

Moles of H_2 = ________________________ mol

d) Then use stoichiometry between Mg and H_2 gas to find the amount of moles of Mg. Then calculate the relative atomic mass of Mg (g/mol)

Relative atomic mass of Mg = ____________________ g/mol

Discussion:

- Discuss possible sources of errors.
- Calculate the % error when compared to the literature value.

Post lab questions:

1. List different metals that can be used in this experiment by giving reasons.
2. Does a piece of Cu wire interfere with the amount of H_2 gas produced? Give reasons.
3. Can you use this experimental set-up to calculate the % of Cu and % of Zn in a piece of brass? Explain briefly.

Experiment 7: Experimental determination of the enthalpy of acid/base neutralization (NaOH and HCl, KOH and HNO_3, NaOH and CH_3COOH, NH_4OH and HCl)

Aims:

1. To learn how to measure temperature changes occurring in chemical reactions.
2. To determine the neutralization enthalpy of acid – base reactions experimentally.

Pre lab questions:

1. Explain how you would estimate the enthalpy of a reaction when the enthalpy values of reactants and products are known.
2. Explain the state function and give some examples.

Introduction:

Most of the physical and chemical changes take place or are carried out under constant pressure conditions. In the laboratory, for example, reactions generally carried out in beakers, flasks, or test tubes that remain open to their surroundings and hence to a pressure of approximately one atmosphere (1 atm, ~10^5 Pa). To quantify the heat flow into or out of a system in a constant-pressure process, chemists use a property called ***enthalpy****,* represented by the symbol *H. i.e. at constant pressure chemist use the relation heat = enthalpy.* Enthalpy is an extensive property; its magnitude depends on the amount of the substance present. It is impossible to determine the enthalpy of a substance, so it is the change in enthalpy, ΔH, that we actually measure.

The ***enthalpy change of reaction,*** ΔH_{rxn} *, is the difference between the enthalpies of the products and the enthalpies of the reactants:*

$$\Delta H_{rxn} = H_{products} - H_{reactants}$$

Heat (*Q*)

As the enthalpy is equal to the heat (*Q*) at constant pressure, we may consider the measurement of heat changes. In the laboratory heat changes in physical and chemical processes are measured with a calorimeter, a closed container designed specifically for this purpose. To estimate heat changes we first need to have an understanding of specific heat and heat capacity.

The specific heat (c) of a substance is the amount of heat required to raise the temperature of one gram of the substance by one degree celsius or kelvin. The heat capacity (C) of a substance is the amount of heat required to raise the temperature of a given quantity of the substance by one degree celsius or kelvin. Specific heat is an intensive property, whereas heat capacity is an extensive property. The relationship between the heat capacity and specific heat of a substance is

$C = m\,c$

Note: sometimes s is used to denote specific heat

Knowing the specific heat c and the amount of a substance, the change in the sample's temperature Δt (temperature in ^{0}C) or ΔT(temperature in K) , the amount of heat (***Q***) that has been absorbed or released in a particular process can be calculated by the equation ;

$Q = m\,c\,\Delta t$ or $Q = m\,c\,\Delta T$

This is exothermic reaction

The enthalpy change is given by ,

$$\Delta H = -\left[\frac{Q}{moles\ of\ the\ reactant\ (limiting)}\right]$$

Note for teachers: Class can be divided into 4 groups to perform experiment on four sets and share the data.

Equipment and chemicals required:

Equipment	Chemicals
Plastic cups (or beakers)(100/200 cm^3)	2.0 mol dm^{-3} NaOH ~ 250 cm^3
250/500 cm3 beaker	2.0 mol dm^{-3} HCl ~ 250 cm^3
Thermometer	2.00 mol dm^{-3} HNO_3 ~ 100 cm^3
Glass rod	2.00 mol dm^{-3} KOH ~ 100 cm^3
	2.00 mol dm^{-3} CH_3COOH~100 cm^3
	2.00 mol dm^{-3} NH_4OH ~ 100 cm^3

Experimental Set-up

Set up the apparatus as described in the following diagram

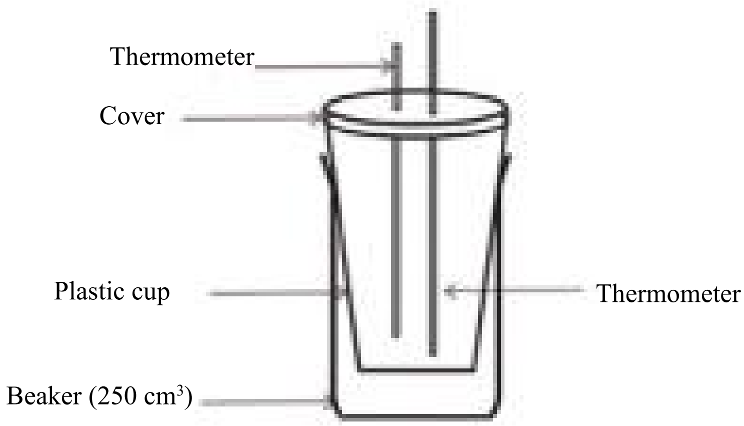


Figure 7.1. Experimental set-up

Procedure

HCl and NaOH

- Place a plastic cup in to a 250 cm^3 beaker as shown in the diagram.
- Add 50.0 cm^3 of 2.00 mol dm^{-3} HCl into the cup and measure the temperature.
- Transfer 50.0 cm^3 of 2.00 mol dm^{-3} NaOH into a beaker and measure the temperature.
- Add the above NaOH solution to a plastic cup containing HCl immediately and record the temperature each 30 second until the maximum value is reached.

Repeat the above procedure for other acid – base couples. Make sure to clean plastic cup, thermometer and glass rod before each experiment.

Experimental results

Reaction: *NaOH (aq) + HCl (aq) → NaCl (aq) +* H_2O *(l)*

Initial temperature of HCl and NaOH / ^{0}C *	Final temperature of solution / ^{0}C	Δt of solution/ ^{0}C

Reaction: *KOH (aq) +*HNO_3*(aq) →* KNO_3 *(aq) +* H_2O *(l)*

Initial temperature of HNO_3 *and KOH* / ^{0}C *	Final temperature of solution / ^{0}C	Δt of solution/ ^{0}C

Reaction: *NaOH (aq) +* CH_3COOH *(aq) →* CH_3COONa *(aq) +* H_2O *(l)*

Initial temperature of CH_3COOH *and NaOH* / ^{0}C *	Final temperature of solution / ^{0}C	Δt of solution/ ^{0}C

Reaction: *HCl (aq) +* NH_4OH *(aq) →* NH_4Cl*(aq) +* H_2O *(l)*

Initial temperature of HCl and NH_4OH / ^{0}C *	Final temperature of solution / ^{0}C	Δ of solution/ ^{0}C

*If the initial temperatures of solutions are different, take the average.

Calculations

- Estimate the amounts (moles) of reactants in each case.
- Estimate the heat liberated during each reactions. The specific heat capacity of water is

  4.2 J g^{-1} K^{-1} and the density of water = 1.000 g cm^{-3} and the volume of solution is 200 cm^3.

$$\boldsymbol{Q = m\ c\ \Delta T}$$

Q = (100 cm^3 x 1.000 g cm^{-3}) x 4.2 J g^{-1} K^{-1} x ΔT = …….. J

Calculate of the ΔH_{neu} for each reactions in kJ mol^{-1} NaOH: Using,

$$\Delta H = \frac{-Q}{\textit{amount of moles of the reactant(limiting)}} = \ldots\ldots\ldots \text{ kJ mol}^{-1}$$

Post lab questions:

1. Discuss the possible sources of errors in this experiment.
2. What is the meaning of negative sign in front of ***Q*** ?
3. Assume that you are provided with 2.00 mol dm^{-3} NH_4OH solution. Without doing an experiment with that solution, explain how you would estimate the enthalpy of the reaction,
   $HCl (aq) + NH_4OH (aq) \rightarrow NH_4Cl(aq) + H_2O(l)$. Compare the value you obtained with the experimentally determined value.

Experiment 8: Validation of Hess Law through experiment

Aims:

1. To determine enthalpy changes of an exothermic reaction carried out using two different pathways of getting from reactants A to products B.
2. To identify the of Hess law by using above system.

Pre lab questions:

1. Define the enthalpy of a reaction.
2. How would you determine the enthalpy of a reaction experimentally?
3. Explain under what condition the heat equals the enthalpy of a reaction.
4. Calculate the heat of solution (ΔH_{soln}) for sodium hydroxide (NaOH) in kJ mol^{-1} for the following observations.
5. When 4.00 g of NaOH was added to a plastic cup containing 100.0 cm^3 of water at 25 ^0C the maximum temperature attained after complete dissolution of NaOH was 35 ^0C. The specific heat capacity of water is 4.2 J g^{-1} K^{-1} and the density of water is 1.000 g $cm^{-3.}$ Na = 23; O = 16;H = 1 (in g mol^{-1}) . Heat absorbed by the plastic cup is negligible.
6. Why the usage of a calorimeter is important in the above type of experiments?

Introduction

In thermochemistry, Hess law is applied to determine the enthalpy of a process which cannot be done as a single reaction in the laboratory. Hess law states that, *" Regardless of the path taken in going from the initial state to the final state, ΔH (or ΔH° if the process is carried out under standard conditions) has the same value or it is independent of the route".* In this experiment, a plastic cup (assuming as insulator) will be used to measure the heat released by three different reactions in which one of the reactions can be expressed as the combination of the other two reactions. Thus, according to Hess law, heat of reaction of a single reaction should be equal to the sum of the heats of reaction for the other two. The objective of this experiment is to measure heats of reactions and to confirm Hess law. The two pathways used in this experiment are:

Path I:

Reaction A. NaOH (s) {10 g} + H_2O (*l*) {125 cm^3} → NaOH (aq) : $\Delta \boldsymbol{H}_1$

Reaction B. NaOH (aq) {2 M, 125 cm^3} + HCl (aq) {2 M, 125 cm^3} → NaCl (aq) + H_2O (*l*) : $\Delta \boldsymbol{H}_2$

Path II:

Reaction C. $HCl\ (aq)\{2\ M,\ 125\ cm^3\} + H_2O\ (l)\{125\ cm^3\} \rightarrow$
$HCl\ (aq)\{1\ M,\ 250\ cm^3\}$: $\Delta \boldsymbol{H_3}$

Reaction D. $NaOH\ (s)\ \{10\ g\} + HCl\ (aq)\ \{1\ M,\ 250\ cm^3\} \rightarrow$
$NaCl\ (aq) + H_2O\ (l)$: $\Delta \boldsymbol{H_4}$

In the above reactions we can see that reactions in paths I and II yield the same result. Therefore, according to Hess law, we should also be able to combine the heat, ***Q*** values for reactions and hence enthalpy.

As these reactions are carried out in an aqueous solution, the energy given off (or taken up) by the system is assumed to be exchanged with water (since the solutions used are dilute, we will assume that they have the same thermal properties as water). For example, if a reaction releases heat (i.e. an *exothermic reaction*), then the temperature of the water will increase. On the other hand, an endothermic reaction will absorb heat from the water, thus causing a decrease in the temperature of water. This allows for a simple calculation of the heat of the reaction by first measuring the temperature change for water, and then using the following equation to calculate the heat, q_{water} absorbed (or given off) by water. We can use the following expression for the calculation of heats of each reactions.

$$\boldsymbol{Q = m\ c\ \Delta T}$$

In this equation ***m*** is the mass of the water (for simplicity, 1.00 cm^3 of water weighs 1.00 g), ***c*** is the specific heat capacity of water, 4.2 J g^{-1} K^{-1}, and $\Delta \boldsymbol{T}$ is the temperature change measured. In this experiment, it will be assumed that no heat lost to the plastic cup/calorimeter (*an assumption that is not entirely true*). We assume that the plastic cup calorimeter is an insulator (i.e. $Q_{calorimeter} = 0$). That is, we are assuming water in the solution gains all the heat lost by the reaction and none is lost to the surrounding or to the cup itself. Thus,

$$Q_{reaction} = -\,Q_{water}$$

Therefore, the enthalpy change ΔH of the reaction is given by,

$$\Delta H = -\left[\frac{Q}{moles\ of\ the\ reactant\ (limiting)}\right]$$

Equipment and chemicals required:

Equipment	Chemicals
• 250/500 cm^3 beaker	• Solid NaOH pellets ~ 40 g
• Thermometer	• 1.00 mol dm^{-3} NaOH~250cm^3
• Glass rod	• 2.00 mol dm^{-3} HCl ~ 500 cm^3
• Balance	• Distilled water
• 2 graduated cylinders (100 cm^3)	
• plastic cup (100/200 cm^3)	

Note for teachers: Class can be divided into 4 groups to perform experiment on four sets and share the data.

Experimental set-up

Set up the apparatus as described in the following diagram

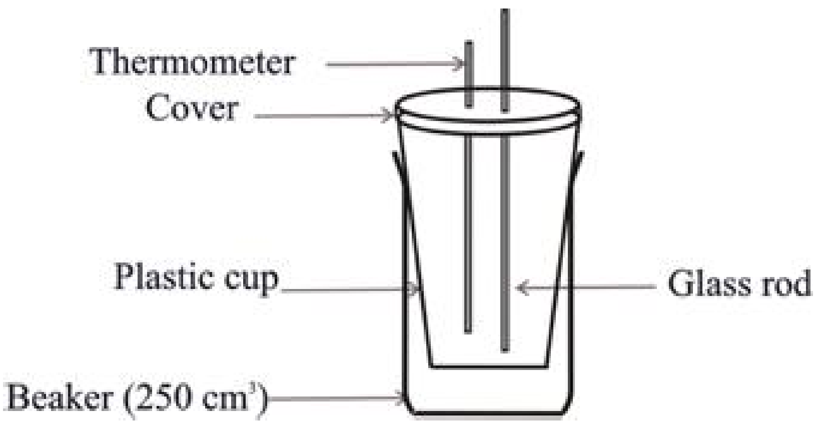


Figure 8.1. Experimental set-up

Procedure:

Path I:

Reaction A. **NaOH (s) {10 g}+ H_2O (l) {125 cm^3} → NaOH (aq) (125 cm^3) : ΔH_1**

i. Place a plastic cup in a 500 cm^3 beaker as shown in the diagram.

ii Add 125.0 cm^3 of distilled water into the cup and measure the temperature of the water till a constant value reaches (t_1).

iii Weigh 10.00 g of solid sodium hydroxide, NaOH, (accurately) and add immediately into the water in the cup described above and stir with a glass rod. Record the temperature each 30 seconds until the maximum value is reached (t_2).

Reaction B. NaOH (aq) {2 M, 125 cm³} + HCl (aq) {2 M, 125 cm³} → NaCl (aq) + H_2O (*l*) : ΔH_2

i. Measure 125.0 cm³ of 2.00 mol dm⁻³ HCl into a beaker and record the temperature (t_3).

ii. Add the the above HCl solution into the cup containing 125.0 cm³ of NaOH solution prepared in **reaction A.** (t_4)

iii. Stir well and record the temperature in each 30 seconds until the maximum value is reached (t_5).

Reaction C. HCl(aq){2 M, 125 cm3} +H_2O (l){125 cm³}→HCl (aq){1 M, 250 cm³} : ΔH_3

i. Rinse well, the cup, thermometer and the glass rod with distilled water.

ii. Measure 125.0 cm³ of 2.00 mol dm⁻³ HCl solution (t_7)and add to the plastic cup containing 125.0 cm³ of distilled water (t_6) and record the temperature(s) (t_8).

Path II

Reaction D. NaOH (s) {10 g} + HCl (aq) {1 M, 250 cm³} → NaCl (aq) + H_2O (*l*) : ΔH_4

i. Weigh 10.00 g of solid sodium hydroxide, NaOH, (accurately) and add immediately into the cup described in above **reaction C.** (t_9)

ii. Stir with a glass rod. Record the temperature each 30 seconds until the maximum value is reached (t_{10}).

Record the results as follows.

Experimental results

Reaction A. NaOH (s) {10 g} + H_2O (*l*) {125 cm³} → NaOH (aq) : ΔH_1

Mass of NaOH	Initial temperature of water / ⁰C (t_1)	Final temperature of solution / ⁰C (t_2)	Δt of solution/ ⁰C $t_2 - t_1$
10.00 g			

Reaction B. NaOH (aq) + HCl (aq) {2 M, 125 cm³}→ NaCl (aq) + H_2O (*l*)) : ΔH_2

Initial temperature of NaOH / ⁰C (t_3)	Initial temperature of HCl / ⁰C (t_4)	Final temperature of solution /⁰C (t_5)	Δt of solution/ ⁰C $t_5 - \{(t_3 + t_4)/2\}$

Reaction C. HCl (aq){2 M, 125 cm³} +H_2O (l){125 cm³}→ HCl (aq){1 M, 250 cm³} : ΔH_3

Initial temperature of H_2O / ^0C (t_6)	Initial temperature of HCl / ^0C (t_7)	Final temperature of solution / ^0C (t_8)	Δt of solution/ ^0C $t_8 - \{(t_6 + t_7)/2\}$

Reaction D. NaOH (s) {10 g} + HCl (aq) {1 M, 250 cm³} → NaCl (aq) + H_2O (*l*) : ΔH_4

Mas of NaOH	Initial temperature of HCl / ^0C (t_9)	Final temperature of solution / ^0C (t_{10})	Δt of solution/ ^0C $t_{10} - t_9$

Calculations

- Estimate the amounts (moles) of HCl and NaOH in each case and find the limiting reagent.
- Estimate the heat liberated during the reactions for each A, B, C and D. The specific heat capacity of water = 4.2 J g^{-1} K^{-1} and the density of water = 1.000 g cm^{-3}.

$$Q = m\,c\,\Delta T$$

$Q = (V\ cm^3 \times 1.000\ g\ cm^{-3}) \times 4.2\ J\ g^{-1}\ K^{-1} \times \Delta T$ = …….. J (V is the total volume of the solution)

Identify the enthalpies associated with each process (step).

a. Calculate of the ΔH_1 for reaction A in kJ g^{-1} NaOH = ………… kJ g^{-1}

and in kJ mol^{-1} NaOH: using,

$$\Delta H_1 = \frac{-Q}{moles\ of\ the\ reactant(limiting)} = \ldots\ldots\ kJ\ mol^{-1}$$

b. Calculate of the ΔH_2 for reaction B in kJ g^{-1} NaOH = ………… kJ g^{-1}

and in kJ mol^{-1} NaOH: using,

$$\Delta H_2 = \frac{-Q}{Asmoles\ of\ the\ reactant(limiting)} = \ldots\ldots\ kJ\ mol^{-1}$$

Likewise,

c. Calculate ΔH_3 for reaction C in kJ mol^{-1} NaOH = ……… kJ mol^{-1}

d. Calculate ΔH_4 for reaction D in kJ mol^{-1} NaOH = ……… kJ mol^{-1}

Demonstration of Hess law: Demonstrate relationship among the above two paths in a manner consistent with Hess law by figuring out an appropriate way. You should notice that

$$\Delta H_1 + \Delta H_2 = \Delta H_3 + \Delta H_4$$

Note: As the assumption made in these experiments to ignore the heat absorbed by the cup calorimeter, there will be an appreciable error in the determination of enthalpy. This can be avoided by determining the heat capacity of the cup calorimeter by simple experiment and incorporating that in the estimation of heats as follows.

Estimation of heat capacity of a cup calorimeter:

Use the same set-up used for the previous experiments.

1. Add 100.0 cm^3 of distilled water into the cup calorimeter and measure the temperature of the water till a constant value is reached (t_{11}).
2. Heat about another 200.0 cm^3 distilled water up to around 80^{0}C in a separate beaker.
3. Measure 100.0 cm^3 of hot water into another beaker and record the temperature (t_{12}).
4. Add immediately the hot water above to the cup calorimeter containing water at room temperature, stir well and record the temperature of the mixture every 30 seconds for about 5 minutes and obtain the constant value (t_{13}).

Calculation:

- Calculate the heat loss (Q_1) by hot water....
  $Q_1 = (100 \text{ cm}^3 \times 1 \text{ g cm}^{-3}) \times 4.2 \text{ J g}^{-1} \text{ K}^{-1} \times (t_{12} - t_{13}) \text{ K} \times (1 \text{ kJ}/ 10^3 \text{ J})$
- Calculate the heat absorbed by calorimeter (Q_c)
  $Q_c = \text{Cc J K}^{-1} \times (t_{13} - t_{11}) \text{ K} \times (1 \text{ K J}/ 10^3\text{J})$
- Calculate the heat absorbed by cold water(Q_2)
  $Q_2 = (100 \text{ cm}^3 \times 1 \text{ g cm}^{-3}) \times 4.2 \text{ J g}^{-1} \text{ K}^{-1} \times (t_{13} - t_{11}) \text{ K} \times (1 \text{ kJ}/ 10^3 \text{ J})$

$(Q_{c\,+}Q_2) = Q_1$ from the equation calculate the Cc.

After the estimation of heat capacity Cc of calorimeter, calculate enthalpy changes of reactions by applying following modification.

Q_{rxn} ***= - [heat absorbed by water + heat absorbed by calorimeter]***

$Q_{rxn} = - [\,(m\,c\,\Delta t) + Cc\,\Delta t]$

Hence, calculate new enthalpy change values in kJ mol^{-1} and compare those with previous values calculated without using heat capacity of the cup calorimeter.

Post lab questions:

1. How would you minimize errors in the experiment?

2. Discuss the deviation of your estimations by comparing with standard enthalpy values, ΔH_{rxn} of the reactions A, B, C and D. Comment on the value of ΔH_3.
3. Use the standard enthalpies of formation, ΔH^0_f, to calculate the "true" (i.e. accepted) ΔH_{rxn} value for all reactions. Perform the calculations for all reactions by considering the aqueous ionic equations for each reaction.
4. You are given the following three reactions. Using them develop an experimental procedure to demonstrate Hess law.
   Reaction P. $NaOH\,(s) \rightarrow NaOH\,(aq)$
   Reaction Q. $NaOH\,(s) + HCl\,(aq) \rightarrow NaCl\,(aq) + H_2O\,(l)$
   Reaction R. $NaOH\,(aq) + HCl\,(aq) \rightarrow NaCl\,(aq) + H_2O\,(l)$

Experiment 9: Comparison of reactions of s block metals with air, water and acids

Aims:

1. To identify reactions of s block elements with air, water, and acids
2. To write balanced chemical reactions for above observations
3. To identify and explain periodic trends observed in group 1 and 2 down the group and across the period

Pre lab questions:

1. Write balanced chemical equations for reactions of s block elements with O_2 in air.
2. Write balanced chemical equations for reactions of s block elements with cold water and hot water
3. Write balanced chemical equations for reactions of s block elements with dil.HCl.

Introduction:

Elements in the s block are those in which last electron is filled into a s orbital in the valence shell. Therefore, only group I and II are considered s block elements in the periodic table. There are variations observed in reactivity of s block elements when considering reactions down the group and across the period with air, water, and acids. These trends are observed experimentally in this laboratory exercise.

Equipment and chemicals required:

Equipment and items	Chemicals
Test tubes Boiling tubes Droppers Glass rods Beakers (400 ml) Bunsen burner Test tube holders Sand papers, filter papers	Elements of s block (Na, Mg, Ca) (0.5g each) dil.HCl or dil.H_2SO_4 (25 ml) Phenolphthalein indicator (few drops)

Precautions:

- Care should be taken when handling sodium metal in reactions could be violent or explosive.
- Never place a piece of sodium metal in acid solution.
- Use protective eye wear and gloves when handling chemicals.

Procedure:

Use the chart below to conduct the experiment and record observations.

Observations and inferences:

	Test	Observation	Inferences with balanced chemical equation
1	Record how s block elements are stored in the laboratory.	Na = Mg =	Reasons:
2	Cut a piece of Na metal and observe changes in the newly cut surface when exposed to air for about 5 min.	New surface : After 5 min.:	
3	Take about 5 cm long Mg metal strip and clean it using a sand paper. Let it exposed to air for 15 min. and record observations.	New surface: After 5 min.:	
4	Heat a piece of magnesium ribbon in air. What do you observe?		
5	Fill a trough half - full with water, add a little of phenolphthalein indicator. Clean a tiny piece of sodium with a filter paper and put it to water. (Wear goggles!) Record observations.		
6	Put a piece of magnesium ribbon cleaned with sand paper into a test tube containing water. Add a little phenolphthalein also. What are your observations?		
7	Put a clean piece of magnesium ribbon in to another test tube containing water and a little phenolphthalein. Heat and observe.		
8	Take a clean piece of magnesium ribbon into a test tube and add dilute Hydrochloric acid. What are your observations?		

Discussion:

- Explain why certain precautions are taken when storing reactive metals like Na in the laboratary.
- Why reaction of sodium metal with dil. acid is not conducted in the laboratary?
- What is the role of phenolphthalein when studying reaction of Mg strip with water? Do you expect the colour of the water to change? Explain.

Post lab questions:

Compare reactivity of group I metals vs. group II metals when exposed to air, water, and acids.

Experiment 10 : Identification of elements in compounds by the flame test (Li, Na, K, Ca, Sr, Ba)

Aims:

1. To test different metal salts using proper methods
2. To identify the metal ions based on the colour of the flame
3. To explain the scientific background of observing different coloured flames based on the metal ion of interest

Pre lab questions:

1. What are the different regions of electromagnetic radiation?
2. What is wavelength range of the visible region in nano meters?
3. What is an atomic spectrum? Explain the origin of different lines observed in the spectra using hydrogen as an example.

Introduction:

Visible light is the region in electromagnetic radiation that we are very familiar with. We see different colours due to differences in wavelengths in the visible region of electromagnetic radiation. When an atom absorbs energy, electrons can jump from a lower to a higher energy level. In the case of flame test, energy necessary for the electronic transition is supplied as heat. When electrons at higher energy levels return to lower energy levels, energy is released as electromagnetic radiation. If this electromagnetic radiation is in the wavelength range of the visible region, we see colours. Since different metals emit radiation in different regions of visible region, we can use characteristic colours observed in the flame as an identification tool.

Equipment and chemicals required:

Equipment	Chemicals
Watch glass Droppers (pasture pipettes) Glass rods Platinum wire / Nichrome wire / Graphite rod Bunsen burner Cobalt glass	Salts containing Li^+, Na^+, K^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+} salts (water soluble salts) Conc.HCl acid

Precautions:

- Care should be taken when handling conc.HCl to avoid burns.
- Platinum wire / Nichrome wire / Graphite rod should be cleaned until no colour is observed on the flame before testing for a specific metal.

Procedure:

- Clean the nichrome or platinum wire by dipping it in conc. HCl and holding it in the Bunsen flame. Repeat until flame gives no colour.
- Dip the cleaned nichrome or Pt wire in the metal salt followed by wetting it with conc. HCl. Then hold the nichrome or Pt wire in the blue region in the Bunsen flame and record the colour of the flame.
- Use the following chart to record your observations. Use colour pencils if available.

Observations and inferences:

	Compound	Flame colour
1	Li_2CO_3	
2	NaCl	
3	Na_2SO_4	
4	Na_2CO_3	
5	KCl	
6	$MgCl_2$	
7	$MgSO_4$	
8	$CaCl_2$	
9	$SrCl_2$	
10	$BaCl_2$	

Post lab questions:

1. Explain why nichrome or Pt wire is cleaned using conc. HCl acid?
2. Explain why nichrome or Pt wire containing a metal salt is wetted with conc. HCl prior to holding it to the Bunsen flame.
3. Which element did not give any colour to the flame?
4. Did you observe the same colour or different colours in the flame for metal salts with different anions but a common cation (eg.NaCl, Na_2SO_4).
5. Why do you use the non-luminous zone in the Bunsen flame for the flame test?
6. Why is it recommended to use a Pt or nichrome wire rather than an iron or Cu wire? Out of Pt and nichrome, which is most recommended?
7. Can you use flame test for a mixture of metal salts with different cations?
8. Name other metal compounds which do not belong to *s* block but give flame colours.
9. How would you use the cobalt glass to distingusih a mixture of K^+ and Na^+ ions. Explain.

Experiment 11: Identification of anions
(SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, S^{2-}, CO_3^{2-}, NO_3^-, NO_2^-,)

Aims : To provide necessary knowledge,

1. to identify SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, S^{2-}, CO_3^{2-}, NO_3^-, NO_2^-**,** in aqueous solution
2. to identify carbon dioxide gas
3. to gain skills in the use of delivery tubes

Pre lab questions:

Complete the following ionic equations.

1. $S_2O_3^{2-}$ (aq) + H^+ (aq)
2. SO_3^{2-} (aq) + H^+ (aq)
3. S^{2-} (aq) + H^+ (aq)
4. NO_2^-(aq) + H^+ (aq)
5. CO_3^{2-}(aq) + H^+ (aq)

Equipment and chemicals required:

Equipment	Chemicals
Test tubes	$AgNO_3$ solution (0.10 moldm^{-3})
Boiling tubes	Na^+ or K^+ salts of SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, S^{2-}, CO_3^{2-}, NO_3^-, NO_2^- (1.0 mol dm^{-3})
Droppers	Dil. NaOH (aq) solution
Glass rods	Dil.HCl acid
Beakers (400 ml)	$BaCl_2$ solution (0.01 mol dm^{-3})
Bunsen burner	Ni^{2+}(aq) and Cu^{2+}(aq) solutions (0.01 mol dm^{-3})
Test tube holders	$Pb(NO_3)_2$ (aq)
Filter papers	Conc.H_2SO_4 acid
Water bath	Fe^{2+}(aq) solutions (1.0 mol dm^{-3})
Red litmus	Aluminum powder
Ice cubes	Conc.NaOH
Conical flask	Conc.HCl
Thermometer	Aniline or p-aminosulphonic acid , phenol
Delivery tube	Na_2CO_3 (water soluble carbonate)
	Lime water
	$CaCl_2$ (aq) solution

Procedure: Use the chart below to conduct the experiment and record observations.

	Test	Observation (colour, precipitate, gas evolved, etc.)	Inferences with balanced chemical equations
Identification of SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, S^{2-} (take about 1 cm^3 of the solution from each anion to a test tube)			
1.	For SO_4^{2-} ions : Add about 1cm3 of $BaCl_2$ solution. Then add dil. HCl.		
2.	For SO_3^{2-} ions : (i) Add a small amount of dil. HCl to about 0.5 g of the solid compound and warm. Hold a filter paper moistened with acidified $K_2Cr_2O_7$ over the mouth of the boiling tube. (ii) Add about 1cm^3 of $BaCl_2$ solution. Then add dil. HCl.		
3.	For S^{2-} ions : (i) Add $AgNO_3$ or $CuSO_4$. solution		
	(ii) Add dil. HCl solution to the solid compound. Hold a filter paper soaked with $Pb(CH_3COO)_2$ solution.		
4.	For $S_2O_3^{2-}$ ions : (i) Add dil. HCl solution and note observations immediately and after some time.		
	(ii) Hold a filter paper moistened with acidified $K_2Cr_2O_7$ over the mouth of the boiling tube		
	(iii) Add $AgNO_3$ solution and warm the solution.		
	(iv) Add $Pb(CH_3COO)_2$ solution and heat the resulting solution.		

	Test	Observation (colour, precipitate, gas evolved, etc.)	Inferences with balanced chemical equations
	Identification of NO_3^-, NO^{2-} (take 1 cm^3 of the solution from each anion to a test tube)		
1.	For NO_3^- ions : Add 3 cm^3 of freshly prepared saturated $FeSO_4$ solution to 2 cm^3 of NO_3^-(aq) solution and then add 3-4 cm^3 of conc. H_2SO_4 slowly down the side of the test tube (Brown ring test.) ring formation is not visible if solution is shaken or the solution is warm.)		
2.	For NO_2^- ions : Add dil. HCl to the solid compound.		
3.	For NO_3^- ions and NO_2^- ions : Take 1 cm^3 of NO_2^- / NO_3^- solution Add Al powder / Devarda's alloy / Zn dust and then conc.NaOH (aq) .Gently warm the solution if necessary. Test the gas liberated with moist red litmus paper or a paper soaked in Nessler reagent.		
	Identification of CO_3^{2-} ions		
1	Add dil. HCl to a solid carbonate (~0.5 g) in a test tube. Pass the liberated gas immediately into the lime water using the delivery tube. Continue passing the excess gas into lime water.		

Post lab questions:

(i) How do you distinguish between the following ions? (Solid compounds are given.)

(i) SO_4^{2-} and SO_3^{2-}

(ii) SO_3^{2-} and $S_2O_3^{2-}$

(iii) NO_3^- and NO_2^-

(ii) Can you use "Brown ring" test as listed above to distinguish a solution of nitrates and a solution of nitrites. Give reasons.

Experiment 12: Showing the presence of nitrogen in air by experiment

Aim:

To demonstrate reactions of Mg with air and formation of Mg_3N_2

Pre lab questions:

1. Write balanced chemical equations for the reactions of a piece of Mg strip when exposed to air and when burned in air.
2. Write balanced chemical equations for the reactions of Mg metal reacting with cold water and hot water.

Introduction:

Air that we breathe is a mixture of several gases. The main constituent in air is nitrogen which contributes for about 79% while oxygen is the second most abundant with nearly 20%. The rest of the gases such as carbon dioxide, inert gases, etc. contribute for about 1%. Therefore, air serves as the main resource to extract nitrogen gas. In this experiment the presence of nitrogen gas in air is demonstrated by forming Mg_3N_2 when a piece of Mg strip is burned in air. Then detecting ammonia gas which is formed upon reaction of Mg_3N_2 with water is used to confirm the formation of Mg_3N_2 and hence, the presence of nitrogen in air.

Equipment and chemicals required:

Equipment	Chemicals
Crucible with lid Clay triangle Droppers(Pasture pipettes) Glass rod Bunsen burner Pair of tongs Sand papers Litmus papers Tripod	Mg strip Nessler reagent

Experimental set-up:

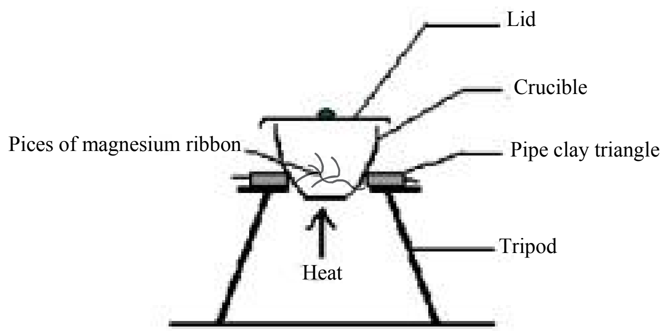


Figure 12.1. Experimental set-up

Precautions:

- Care should be taken when handling the crucible.
- Never look directly into the crucible with burning Mg strip.
- Use protective eye wear and gloves.

Procedure:

- Clean a piece of Mg strip with a sand paper.
- Cut it into very small pieces.
- Place the Mg pieces in the crucible and close it with lid and heat for about ½ an hour. Do not open the lid while heating.
- Allow the crucible to cool to room temperature.
- Add a little of aqueous Nessler reagent to the white residue.
- Observe any colour changes in the solution.

Results:

1. What is the colour change observed when aqueous Nessler's reagent is added to the residue?

   ……………………………………………………………………………………

2. Identify the gas evolved based on the above results.

   ……………………………………………………………………………………

Post lab questions:

1. Why do you need to cleam the piece of Mg strip before starting the experiment?
2. Write the balanced chemical equation for the reaction of Mg_3N_2 with water. What is the colour change observed when the wet red litmus paper is held to the evolving gas of this reaction ?

Experiment 13: Identification of halides

Aim:

To develop the skill of identifying chloride, bromide and iodide ions when they exist separately in solution.

Pre lab questions:

Write balanced chemical equations for the reactions of chloride, bromide and iodide ions with chlorine water, silver nitrate and lead acetate.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes	Na^+ or K^+ or NH_4^+ salts of Cl^-, Br^-, I^- (0.10 $moldm^{-3}$ solutions)
Boiling tubes	$AgNO_3$ solution (0.10 $moldm^{-3}$)
Droppers (Pasture pipettes)	Conc.H_2SO_4 acid
Glass rods	$Pb(NO_3)_2$ (aq) or $Pb(CH_3COO)_2$ (aq)
Beakers (400 ml)	Chlorine water
Bunsen burner	Chloroform or carbon tetrachloride
Test tube holders	Dil. NH_4OH solution (1.0 $moldm^{-3}$)
Filter papers	Dil.HNO_3 acid
Water bath	Starch solution
Litmus papers	

Procedure:

Use the chart below to do the following tests separately for chlorides, bromides and iodides and record observations.

Test	Observation	Inferences with balanced chemical equations
Test 1: Take about 1 cm^3 of halide solutions separately into test tubes.		
For chlorides: (i) Add silver nitrate solution, a little at a time and observe whether precipitation occurs. Add dil. HNO_3 acid (ii) Add dil. ammonia solution to these precipitates and note down the observations.		

Test	Observation	Inferences with balanced chemical equations
For bromides: (i) Add silver nitrate solution, a little at a time and observe whether precipitation occurs. Add dil. HNO_3 acid (ii) Add ammonia solution to these precipitates and note down the observations.		
For iodides: (i) Add silver nitrate solution, a little at a time and observe whether precipitation occurs. Add dil. HNO_3 acid (ii) Add dil. ammonia solution to these precipitates and note down the observations.		
Test 2: Take halide solutions separately into test tubes.		
For chlorides: (i) Add $Pb(CH_3COO)_2$ solution to chloride solution in a test tubes. Observe the colour of the precipitate formed. (ii) Dilute the precipitates with water and heat, then allow to cool again.		
For bromides: (i) Add $Pb(CH_3COO)_2$ solution to bromide solution in a test tube. Observe the colour of the precipitate formed. (ii) Dilute the precipitates with water and heat, then allow to cool again.		
For iodides: (i) Add $Pb(CH_3COO)_2$ solution to iodide solution in a test tubes Observe the colour of the precipitate formed. (ii) Dilute the precipitates with water and heat, then allow to cool again.		

Test	Observation	Inferences with balanced chemical equations
Test 3: Take halide solutions separately into test tubes.		
For chlorides: Add a few drops of $CHCl_3$ and then add a little chlorine water. Shake the tubes well and observe the colour of the organic layer.		
For bromides: Add few drops of $CHCl_3$ and then add a little chlorine water. Shake the tubes well and observe the colour of the organic layer.		
For iodides: Add few drops of $CHCl_3$ and then add a little chlorine water. Shake the tubes well and observe the colour of the organic layer.		
Note: Use dilute sodium hypochlorite solution acidified with hydrochloric acid can also be used as chlorine water		

Post lab questions

(i) Write balanced equations for the reactions that could occur when solid NaCl, NaBr and NaI are heated with conc. H_2SO_4.

Experiment 14: Standardization of a solution of thiosulphate ions using KIO_3 and KI

Aim:

To provide necessary knowledge to conduct redox titrations.

Pre lab questions:

1. What are the essential characteristics of a compound in order to use it as a primary standard?
2. List chemical compounds that are commonly used as a primary standard.

Introduction:

Solutions of sodium thiosulphate are conveniently standardized by titration of the iodine produced when an excess of potassium iodide is added to a known volume of an acidified standard potassium iodate solution.

$$IO_3^- (aq) + 5I^- (aq) + 6H^+ (aq) \rightarrow 3I_2 (aq) + 3H_2O (aq)$$

Iodine, is ordinarily titrated with a standard sodium thiosulphate solution, with starch serving as the indicator:

$$2\ S_2O_3^{2-} (aq) + I_2 (aq) \rightarrow S_4O_6^{2--} (aq) + 2I^- (aq)$$

Since the concentration of standard potassium iodate solution is known the amount of iodine can be calculated. Then, combined with the titration reading the concentration of the thiosulphate solution can be calculated.

Equipment and chemicals required:

Equipment	Chemicals
Titration flasks	Dil.H_2SO_4 acid (1 mol dm^{-3})
Glass rods	water
Funnel	Starch indicator
Burette	Standardized KIO_3 solution (0.02 mol dm^{-3})
Pipette	KI solution (0.5 mol dm^{-3})
White tile	$K_2S_2O_3$ solution (approximately 0.5 mol dm^{-3})

Experimental set-up:

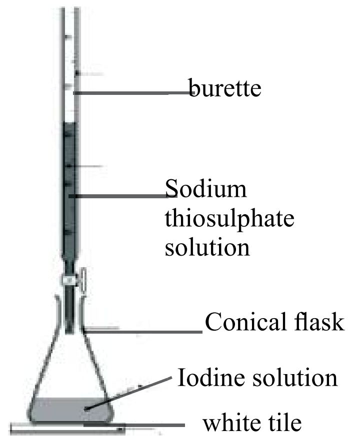


Figure 14.1. Experimental setup

Precautions:

Make sure to use dry KIO_3 when the preparing standard solution.

Procedure:

- Rinse the 25 cm^3 pipette with KIO_3 solution. Then transfer 25.0 cm^3 of KIO_3 solution in to a titration flask.
- Add 20 cm^3 of dilute sulfuric acid, followed by 10 cm^3 of KI solution.
- Rinse the burette with $K_2S_2O_3$ solution and fill it up to the mark.
- Place the titration flask on the white porcelain tile and slowly add $K_2S_2O_3$ solution until the colour of the contents in the titration flask is pale yellow.
- Add a few drops of starch solution and observe the formation of deep blue colour solution.
- Continue addition of $K_2S_2O_3$ solution drop wise until the solution turns colourless at the end point. Record the burette reading and repeat the titration three times.

Results:

	Trial 01	Trial 02	Trial 03	Average
Burette reading (cm^3)				

Calculations:

(i) Calculate the amount of moles of I_2 produced when KI reacted with KIO_3 solution.

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(ii) Calculate the amount of moles of $S_2O_3^{2-}$ ions in the solution titrated.

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(iii) Calculate the concentration of the $S_2O_3^{2-}$ - ions in the solution.

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Post lab questions:

1. What is the role of dil.H_2SO_4 in this experiment?
2. Why starch indicator is added close to the end-point?
3. Which of the three pieces of titration apparatus, the pipette, the burette or the conical flask, should not be rinsed with the solution it is to contain? Give reasons.
4. Why can't you use hydrated KIO_3 for this experiment?
5. Give an alternative method to generate iodine instead of using KIO_3 solution.

Experiment 15: Identification of ammonia gas and ammonium salts

Aim:

To provide necessary knowledge to identify the presence of ammonia gas and ammonium salts.

Pre lab Questions:

1. List chemical reactions that can produce ammonia gas.
2. List household items that have ammonia or ammonium compounds that can liberate ammonia.

Introduction:

Ammonia is a nitrogen containing compound that is produced by nature and humans as well. It is a colourless gas with very irritant and characteristic smell. Ammonia is very soluble in water producing a basic solution. In nature, ammonia is produced by bacteria, decaying plant and animal matter, and animal waste. Industrially, ammonia is produced by the Haber process and it has diverse range of applications such as refrigerant, fertilizers, explosives, dyes, cosmetics, cleaning agents, etc.

The presence of ammonia can be detected by a wet red litmus paper due to formation of weakly basic ammonium hydroxide when dissolved in water. Also, Nessler reagent (K_2HgI_4) is used to confirm the presence of ammonia gas and ammonium salts by the resulting dark brown colouration or precipitate. Often in the laboratory, formation of white smog of NH_4Cl when ammonia gas is exposed to conc. HCl is used as an identification test for ammonia.

Equipment chemicals required:

Equipment	Chemicals
Test tubes	Conc.NH_4OH
Boiling tube	$(NH_4)_2CO_3$ solid
Delivery tube	NH_4Cl solid
Bunsen burner	Conc.HCl
Glass rods	Nessler reagent
Spatula	
Litmus papers	
Filter papers	

Precautions: Ammonia is an irritant gas; therefore, do not inhale and avoid contact with skin and eyes. Also care should be taken when handling concentrated acids to avoid burnings. Nessler reagent is toxic due to the presence of mercuric salts; avoid contact with skin and wash hands before leaving the lab. Concentrated ammonia bottle should be opened in an area with good ventilation.

Procedure: Use the chart below to conduct the experiment and record observations.

	Test	Observations			
		Wet red litmus and blue litmus	**A glass rod moistened with**	**Filter paper wetted with Nessler's reagent or adding few drops**	**Inferences and chemical reactions**
1	Hold filter paper dipped in with Nessler reagent, a glass rod moistened with conc.HCl and litmus papers at the mouth of the conc. ammonia bottle.				
2	Place small amount of ammonium salt in a boiling tube and add aqueous NaOH. Test the gas evolved with filter papers with dipped Nessler reagent, glass rod dipped in conc. HCl and litmus papers.				
3	Make a diluted solution of ammonia and test with litmus papers and a few drops of Nessler reagent.				
4	Dissolve a small amount of solid NH_4Cl in water and test with litmus papers and a few drops of Nessler reagent.				

Post lab questions:

1. Why is it necessary to wet the litmus papers when detecting ammonia gas?
2. Which of the following will release ammonia gas upon heating? Explain with balanced chemical equations. (a) $(NH_4)_2CO_3$ (b) NH_4NO_3 (c) NH_4NO_2 (d) $(NH_4)_2Cr_2O_7$
3. Write the balanced equations for the following reactions.
   (a) $NH_4Cl + NaOH \rightarrow$
   (b) $(NH_4)_2CO_3 + KOH \rightarrow$
4. What are the chemicals used to prepare Nessler reagent?

Experiment 16: Testing the solubility of salts of s block elements

Aim:

To provide necessary skills and knowledge to identify salts of s block elements that precipitate in water with common anions

Pre lab Questions:

1. Write chemical formula of s block elements with following anions (halides, OH^-, CrO_4^{2-} , PO_4^{3-}, $C_2O_4^{2-}$,SO_4^{2-}, SO_3^{2-}, S^{2-}, CO_3^{2-}, HCO_3^- NO_3^-, NO_2^-)

Introduction:

It is important to study the solubility of cations of s block elements with different anions to understand periodic trends and differences between elements.

Equipment chemicals required:

Equipment	Chemicals
Test tubes Boiling tube Droppers (pasture pipettes) Glass rods Beakers (400 ml)	0.1 mol dm-3 solutions of **K^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}** salts (water soluble salts) 1.0 mol dm-3 solutions of Na^+ salts of following anions *halides,* CrO_4^{2-} , $PO_4 3^-$, $C_2O_4^{2-}$,SO_4^{2-}, $SO_3 2^-$, S_2^-, CO_3^{2-}, HCO_3^-, NO_3^-, NO_2^-, OH^-

Procedure:

- Prepare above mentioned solutions enough for all students in the class.
- Take 1 ml of each cation solution in to separate a test tubes.
- Add Na^+ salt of each anion drop wise while swirling according to following chart
- Record any coloration or precipitation in the chart.

Observations:

0.1 $moldm^{-3}$ cation solution (1ml)	1 $moldm^{-3}$ NaCl (drop wise)	0.1 $moldm^{-3}$ NaBr (drop wise)	0.1 $moldm^{-3}$ NaI (drop wise)	0.1 $moldm^{-3}$ NaOH (drop wise)	0.1 $moldm^{-3}$ Na_2S (drop wise)	0.1 $moldm^{-3}$ $NaNO_3$ (drop wise)	0.1 $moldm^{-3}$ $NaNO_2$ (drop wise)
K^+							
Mg^{2+}							
Ca^{2+}							
Sr^{2+}							
Ba^{2+}							

Observations:

0.1 $moldm^{-3}$ cation solution (1ml)	1 $moldm^{-3}$ Na_2CO_3 (drop wise)	0.1 $moldm^{-3}$ $NaHCO_3$ (drop wise)	0.1 $moldm^{-3}$ Na_2SO_4 (drop wise)	0.1 $moldm^{-3}$ Na_2SO_3 (drop wise)	0.1 $moldm^{-3}$ $Na_2C_2O_4$ (drop wise)	0.1 $moldm^{-3}$ Na_3PO_4 (drop wise)	0.1 $moldm^{-3}$ $NaCrO_4$ (drop wise)
K^+							
Mg^{2+}							
Ca^{2+}							
Sr^{2+}							
Ba^{2+}							

Post Lab Questions:

1. Identify the trend in solubility of compounds in group I elements
2. Explain the trend in solubility observed for group II cations with hydroxides with carbonates and sulphates by using hydration enthalpy and lattice enthalpy.
3. List any colored precipitates formed and explain the origin of colour (cation based or anion based)

Experiment 17: Testing the thermal stability of nitrates and carbonates bicarbonates of s block elements

Aim:

To provide necessary knowledge to find a pattern in the thermal stability of the nitrates and carbonates bicarbonates of the s block elements

Pre lab questions:

1. What is the chemical compounds in baking powder and washing soda?
2. Find the decomposition temperatures of carbonate, bicarbonate and nitrate compounds in literature and predict the relative trend of thermal stability expected.

Introduction:

Group II bicarbonates do exist in solid states beacuse of their less termal stability

Note : use $NaHCO_3$ only

Equipment chemicals required:

Equipment	Chemicals
Test tubes	Na_2CO_3, $NaHCO_3$, $NaNO_3$
Boiling tubes	K_2CO_3, KNO_3
Litmus papers	$MgCO_3$, $Mg(NO_3)_2$
Glass rods	$CaCO_3$, $Ca(NO_3)_2$
Bunsen burner	$SrCO_3$, $Sr(NO_3)_2$
Test tube holders	$BaCO_3$, $Ba(NO_3)_2$

Precautions: Care should be taken to swirl the boiling tube in the flame rather holding it for a long time. Also when using the delivery tube, make sure to remove the side of lime water first followed by taking the boiling tube away from the flame to avoid lime water going in to the boiling tube.

Procedure: Use the chart below to conduct the experiment and record observations.

	Test	Observation	Inferences with balanced chemical reactions
	Test 1: Carbonates and bicarbonates: Take about 1g of solid carbonate/ bicarbonate in to a boiling tube and fix a stopper carrying a delivery tube. Insert the free end of the delivery tube into about ½ cm height of limewater in a test tube. Heat the boiling tube. Record any gas evolving and a change in color of limewater inside the test tube. Note relative time taken to evolve gas.		

	Test	Observation	Inferences with balanced chemical reactions
1	Na_2CO_3		
2	$NaHCO_3$		
3	K_2CO_3		
4	$MgCO_3$		
5	$CaCO_3$		
6	$SrCO_3$		
7	$BaCO_3$		
	Test 2: Nitrates : Take each nitrate into a boiling tube and heat. Hold a glowing splinter at the mouth. If a colored gas is liberated, compare the time taken for the liberation of the coloured gas. If a colourless gas liberated, measure the time taken for the glow of the splint.		
1	$NaNO_3$		
2	KNO_3		
3	$Mg(NO_3)_2$		
4	$Ca(NO_3)_2$		
5	$Sr(NO_{3)2}$		
6	$Ba(NO_3)_2$		

Post lab questions:

1. Discuss relative thermal stability of Group I and Group II carbonates, bicarbonates, and nitrates using results obtained and with reference to the structural properties.
2. Discuss uses of some of the above compounds in day today life.
3. Write balanced equations for all deposition reactions.

Experiment 18: Identification of the colours of complex ions in aqueous medium

Aim :

Identify the colours of the ions that form by the d block elements in the aqueous medium

Pre lab qcuestions:

1. What is the colour of the $CuSO_4$ aqueous solution
2. Write the formula of the complex cation present in that aqueous solution
3. Some of the d block metal ions show colours in the aqueous solution, but some of them doesn't show the colours. Explain the reason by giving examples.

Introduction:

The characteristic features of the compounds formed by the d block elements is that their aqueous solutions are coloured. If the ions formed by the d block elements have halfly filled d orbitals, they are referred to as transition elements. The characteristic feature of the transition metal ions is that they show colours in aqueous solutions. These ions absorb the selected wave lengths belonging to the white light and show relevant colours at the excited state.

In considering 3d elements, as Zn and Sc do not form ions having unpaired electrons, their aqueous solutions are not coloured. Here H_2O acts as a ligand and form dative bonds with metal cations.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Glass rods Measuring cylinder (10 cm^3)	$CrCl_3$ $MnSO_4$ $FeCl_3$, $FeSO_4$ $Co(NO_3)_2$, $ZnSO_4$ $CuSO_4$, $Ni(NO_3)_2$ Distilled water

Procedure:

- Weigh about 0.5 g of the above mentioned chemical compounds and put them into separate test tubes and label them.
- Add about 10 cm^3 of distilled water to each of the test tubes and dissolved the compound well using a glass rod'
- Record the colours of the aqueous solutions in the following table.

Compound	**Ion**	**Colour of the aqueous solutions**
$CrCl_3$	Cr^{3+}	
$MnSO_4$	Mn^{2+}	
After washing rust, dissolve Fe powder in dil. HCl	Fe^{2+}	
$FeCl_3$	Fe^{3+}	
$Co(NO_3)_2$	Co^{2+}	
$CuSO_4$	Cu^{2+}	
$NiNO_3$	Ni^{2+}	
$ZnSO_4$	Zn^{2+}	

Post lab questions:

1. Give the chemical formula and the structure of the aqueous complex of the above ions.
2. Write their IUPAC names.
3. (a) Name the ion that form colourless aqueous solution.
   (b) Explain the reason for the formation of colourless aqueous solution using the electron configuration.

Experiment 19: Determination of the concentration of a ferrous ion solution using acidified potassium permanganate

Aims:

To provide necessary knowledge

1. on self-indicator titration
2. to prepare a given volume of Fe^{2+} ion solution using a volumetric flask
3. to determine the concentration of Fe^{2+} ion solution using redox titration

Pre lab questions:

1. What is the colour of Fe^{3+} ions and Fe^{2+} ions in aqueous medium?
2. Explain how to prepare 250 cm^3 of a 0.02 mol dm^{-3} $KMnO_4$ solution in the laboratory.

Introduction:

When colored compounds are involved in redox titrations, the titrand and titrant or one of the species may act as the indicator to obtain the end point of the titration. The reactions involved titrating Fe^{2+} ions against MnO_4- ion solution are given below.

$$MnO_4^- (aq) + 8H^+ (aq) + 5e \rightarrow Mn^{2+} (aq) + 4 H_2O(l)$$

$$Fe^{2+} (aq) \rightarrow Fe^{3+} (aq) + e$$

At the end point all Fe^{2+} ions in the solutions are oxidized to Fe^{3+} ions. Hence, further addition of $KMnO_4$ will results in purple colouration. (In dilute solutions of $KMnO_4$ this may appear as pink colouration)

Equipment and chemicals required:

Equipment	Chemicals
Titration flasks Volumetric flask (100 cm^3) Burette Pipette White porcelain tile	$FeSO_4$ solid (fresh pack)/ ferrous ammonium sulphate $KMnO_4$ solution (0.02 $moldm^{-3}$) H_2SO_4 acid (2 $moldm^{-3}$) Conc.H_3PO_4 acid Distilled water

Procedure:

- **Preparation of Fe^{2+} ion solution:** Measure about 3.5 g of $FeSO_4$ transfer it in to a 100 cm^3 volumetric flask. Add 50 cm^3 of 2 $moldm^{-3}$ H_2SO_4 acid in small quantities while swirling until all salts are completely dissolved. Then add distilled water up to the 100 cm^3 mark of the volumetric flask and mix evenly.
- Transfer 25.00 cm^3 of Fe^{2+} ion solution in to a titration flask. Add 25 cm^3 of 2 $moldm^{-3}$ H_2SO_4 acid and 5cm^3 of conc. H_3PO_4 acid.

- Titrate the Fe^{2+} ions in the titration flask against $KMnO_4$ solution in the burette until colour of the solution in the titration flask turn to a pink colour which is stable for more than 30 seconds. Record the burette reading at this point.
- Experiment should be repeated three times until the difference between burette readings at the end points vary by less than 0.1 cm^3. Then the average reading should be taken for the calculation.

Results:

	Trial 01	Trial 02	Trial 03	Average
Volume of $KMnO_4$ used (cm^3)				

Calculations:

(i) Write the balanced redox reaction for this titration.

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(ii) Calculate the moles of $KMnO_4$ used in this titration.

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(iii) Calculate the concentration of Fe^{2+} ion solution

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Post lab questions:

1. How would you know the $FeSO_4$ container is a fresh pack?
2. Why dil.H_2SO_4 acid is added when preparing Fe^{2+} ion solution?
3. What is the role of conc.H_3PO_4 in this titration?
4. Discuss possible sources of errors in this experiment
5. Explain the term “self -indicator” according to this experiment.
6. How would you modify this experiment for a mixture of Fe^{2+} and Fe^{3+} ion solution to find concentration of each species?

Experiment 20: Determination of the concentration of $KMnO_4$ solution using standard acidified $K_2C_2O_4$ solution.

Aims:

To provide necessary knowledge

1. to prepare a known concertation of a primary standard solution
2. to determine the concentration of MnO_4^- ion solution using redox titration

Pre lab questions:

1. Explain why $K_2C_2O_4$ is used as a primary standard?
2. List the qualities of a primary standard.
3. Name the primary standards that you know.
4. What is the most stable oxidation state of Mn in acidic medium?

Introduction:

Potassium permanganate is a strong oxidizing agent. It has intense dark purple colour due to MnO_4^- ions which turn to colourless when reduced to Mn^{2+} ion. This colour change is used in many instances to determine the end point of a titration. Since, $KMnO_4$ is not a primary standard, it is important to titrate the solution of MnO_4^- with a primary standard like $Na_2C_2O_4$ to find the actual concentration. The redox reaction involved in this experiment are given below.

$$MnO_4^-(aq) + 5C_2O_4^{2-}(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

Equipment and chemicals required:

Equipment	Chemicals
Titration flasks	$Na_2C_2O_4$ solution (0.050 moldm^{-3})
Volumetric flask (100 cm^3)	$KMnO_4$ solution (about 0.02 moldm^{-3})
Burette	H_2SO_4 acid (2 moldm^{-3})
Pipette	Distilled water
White porcelain tile	
Watch glass	

Procedure:

- **Preparation of standard $K_2C_2O_4$ (or oxalic acid in case $K_2C_2O_4$ is not available):** Calculate and measure accurately the amount of $Na_2C_2O_4$ or $H_2C_2O_4$ required to prepare 250 cm^3 solution of 0.050 moldm^{-3} concentration. Make sure to use a clean and dry watch glass. Transfer the contents in to the 250 cm^3 volumetric flask using a funnel. Add small quantity of distilled water and swirl to dissolve all salts completely. Then add distilled water up to the 250 cm^3 mark to prepare 0.050 moldm^{-3} solution of oxalic ions.

- Titration: Measure 25.00 cm^3 volume of oxalate ion solution in to the titration flask using a pipette. Add 15 cm^3 of dil.H_2SO_4 acid. Warm the solution. Then fill the burette with $KMnO_4$ solution and continue titration until contents of the titration flask turn to pink color stable for more than 30 seconds. Since this reaction progress slowly at room temperature, make sure to keep warm the contents of the titration flask while adding $KMnO_4$ solution. Repeat titration three times until the difference between burette readings at the end points vary by less than 0.1 cm^3. Then the average reading should be taken for the calculation.

Results:

	Trial 01	Trial 02	Trial 03	Average
Volume of $KMnO_4$ used (cm^3)				

Calculations:

Calculate the concentration of MnO_4^- ion solution

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Post lab questions:

1. Discuss possible sources of errors in this experiment and list suggestions to improve accuracy.
2. Can you use reaction with $Na_2C_2O_4$ to standardize $K_2Cr_2O_7$ solution? Explain.

Experiment 21: Observing the colours of Cu(II), Ni(II) and Co(II) salts with hydrochloric acid and ammonia solution

Aims:

To provide necessary knowledge

1. to explain the fact that *d* block elements can form coordination complexes
2. to prepare a few complex compounds.
3. to state that the hydrated ions of those complex compounds are coloured.

Pre lab questions:

1. Write electron configurations of Cu(II), Ni(II) and Co(II) ions
2. Give reasons why *d* block metals can form complexes?

Introduction:

Elements in groups 3 to 12 are collectively classified as *d* block element. Aqueous solutions of many transition metal ions can absorb radiation in the visible region of the electromagnetic spectrum to produce various colours. This ability is due to the presence of partially filed 3*d* sub shells of these elements. However, ions with d^0 or d^{10} configuration are colourless in an aqueous solution. The presence of partially filed 3*d* sub shells of these elements has the ability to accept lone pairs of electrons to form complex ions. The species that can donate electrons (Lewis base) to the central metal ion (Lewis acid) are called "ligands". The ligands that are coordinated to the central metal ion play a major role in determining the overall colour of the complex. In this experiment, formation and colours of Cu(II), Ni(II) and Co(II) ions with H_2O, NH_3 and Cl^- ions is observed.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Glass rods	Dil.$CuSO_4$ solution Solution with Ni^{2+} ion solution Solution with Co^{2+} ion solution Conc.HCl acid Dil. and conc. NH_3 solution

Procedure: To 1cm^3 of each metal ion add conc. HCl and ammonia solution. Use the chart below to record colour of the solution.

Metal ion	Colour of the aqueous solution	With few drops of dil. NH_4OH	With few drops of conc.	NH_3OH With excess of conc. HCl
Cu^{2+}				
Ni^{2+}				
Co^{2+}				

Post lab questions:

1. Write the chemical formula and structure of complex ions of above metal ions.
2. Name those complex ions according to the IUPAC.

Experiment 22: Experimental observations of different oxidation states (+2,+4,+6,+7) of manganese containing compounds using \ redox reactions

Aim:

To observe colours of Mn compounds in different oxidation states and study how to interconvert between oxidation states.

Pre lab questions:

1. Write the electron configurations of Mn in compounds with different oxidation states.
2. What is the difference between oxidations states observed for MnO_4^- ion reactions at acidic medium vs. basic medium? Give example reactions to support your answer.

Introduction:

Manganese is a *d* block element with electron configuration [Ar] $3d^5$ $4s^2$. Therefore, oxidation state of Mn is observed between 0 to +7. Interestingly, when oxidation states are different the colour of the Mn compounds varies drastically. The use of appropriate reducing agent can control the resulting oxidation state of Mn compound.

The Mn(VII), which is present in an aqueous solution of $KMnO_4$ is purple in colour Concentrated potassium hydroxide converts +7 oxidation state of manganese to +6 state, which is green in colour.

$$2KMnO_4(aq) + 2KOH(aq) \rightarrow 2K_2MnO_4(aq) + H_2O(l)$$

Hydrogen peroxide reduces K_2MnO_4 to MnO_2 which contain brown colour Mn(IV).

When conc. HCl is added to MnO_2, it gets converted to Mn^{2+} ions. Mn^{2+} ions are almost colourless.

$$MnO_2(s) + 4HCl(aq) \rightarrow MnCl_2(aq) + 2H_2O(l) + Cl_2(g)$$

So, different oxidation states of manganese exhibit different colours.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Glass rods	Dil.$KMnO_4$ solution Dil.H_2SO_4 acid Conc.NaOH solution / NaOH solid H_2O_2 solution Conc.HCl acid

Procedure:

- Use the chart below to conduct the experiment and record colour of the solution.

	Test	Observation	Inferences and chemical reactions
1	Take about 2 cm^3 of a dilute potassium permanganate solution to a boiling tube and record the colour of the solution.		
2	Place about 2 cm^3 of a dilute potassium permanganate solution to a boiling tube, acidify slightly with dilute sulphuric acid and add concentrated potassium hydroxide solution till there is a colour change. Record the colour change.		
3	Add about two drops of hydrogen peroxide to the solution. Observe the colour change.		
4	Add concentrated hydrochloric acid to the solution and observe the changes in colour.		

Post lab questions:

1. Write balanced half ionic equations for each step of the experiment.
2. Write the balanced chemicals reactions for reactions of MnO_4^- ions with following reducing agents.
   (a) H_2O_2 (in acidic medium)
   (b) H_2S (in basic medium)
   (c) SO_2 (in acidic medium)

Experiment 23: Tests to identify Ni^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} and Cr^{3+} ions using NaOH and NH_3

Aims:

1. To identify the presence of Ni^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} and Cr^{3+} ions in aqueous medium.
2. To explain identification tests using differences in solubility of the compounds
3. To identify the complex formation by certain cations
4. To identify the colours of d block elements cations

Pre lab questions:

1. Write the electron configurations of Ni^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} and Cr^{3+} ions
2. What are the water soluble compounds of Ni^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} and Cr^{3+} ions?

Introduction:

The differences in solubility of hydroxides and colours formed before and after heating provide information to distinguish between these ions. Furthermore, Ni^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} and Cr^{3+} ions form colored complexes with ammonia which also provide vital information for identification.

Chemicals and items required:

Glassware and items	Chemicals
Test tubes Glass rods Bunsen burner	Aqueous solutions of Ni^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+} and Cr^{3+} ions (0.1 $moldm^{-3}$) Dil.H_2SO_4 acid (2 $moldm^{-3}$) NaOH solution(4 $moldm^{-3}$) NH_4OH solution (4 $moldm^{-3}$) Aqueous solutions of $K_3Fe(CN)_6$ $K_4Fe(CN)_6$, KSCN/NH_4SCN, H_2O_2, dimethylglyoxime (DMG)

Procedure:

- Use the chart below to conduct the experiment and record colour of the solution. Use 1 cm^3 aqueous solution of each metal ions in to separate test tubes to conduct the practical.

	Test	Observations				
		Ni^{2+}	Fe^{2+}	Fe^{3+}	Cu^{2+}	Cr^{3+}
1	(i) Add NaOH dropwise and record any precipitation					
	(ii) If precipitate observed, continue addition of NaOH and record observations					
		Ni^{2+}	Fe^{2+}	Fe^{3+}	Cu^{2+}	Cr^{3+}
2	(i) Add NH_4OH solution dropwise and record any precipitation and colour of solution					
	(ii) If precipitate observed, continue addition of NH_4OH and record observations					
3	Add few drops of $K_3Fe(CN)_6$ solution					
4	Add few drops of $K_4Fe(CN)_6$ solution					
5	Add few drops of KSCN/ NH_4SCN solution					
6	Add NaOH 1cm^3 and H_2O_2 solution					
7	Add DMG solution					

Post lab questions:

1. Write balanced chemical reactions to explain above observations
2. If few drops of H_2O_2 solution is added to alkaline Cr^{3+} solution, explain the observation you expect with balanced chemical reactions.
3. Write the formula of complex ions and name them according to the IUPAC rules.

Experiment 24: Observation of reactions and properties of Alkene and Alkyne.

Aim:

To observes the reactions and properties of ethene and ethyne.

Pre lab questions:

1. Discuss the differences between saturated and unsaturated hydrocarbons
2. What structural feature that distinguish whether a hydrocarbon is an, (a) alkane, (b) alkene, (c) alkyne and (d) aromatic.
3. What are the characteristic reactions of alkyne?
4. Given alkyne (a) is an internal alkyne and alkyne (b) is a terminal alkyne. Propose methods to distinguish such alkynes from one another.

$H_3C—C\equiv C—CH_3$

(a)

$H_3C—CH_2C\equiv CH$

(b)

Introduction:

An atom or a group of atoms in a family of compounds that decide the chemical and physical properties of those compounds are known as a functional group. The simplest type of organic compounds contains hydrogen and carbon, and these organic compounds are called hydrocarbons.

Chemicals and items required:

Glassware and items	Chemicals
Boiling tube	Ethanol
Water bath	Alumina (aluminium oxide)
Delivery tube	calcium carbide
Bunsen burner	Alkaline potassium permanganate
Cotton wool	Acidified potassium permanganate
	Ammonical cuprous chloride

Precautions:

- Assume the organic compounds are highly flammable. Use only small quantities. Keep away from open flames. Assume the organic compounds are toxic and can be absorbed through the skin.

Experimental Setup:

The reaction of alkene will be observed on ethene prepared in the laboratory by the dehydration of ethanol using alumina at high temperature.

$$H_3C-CH_2OH \xrightarrow[\Delta]{\text{Alumina}} H_2C=CH_2$$

The apparatus for the preparation of ethene is shown in the Figure 24.1 given below.

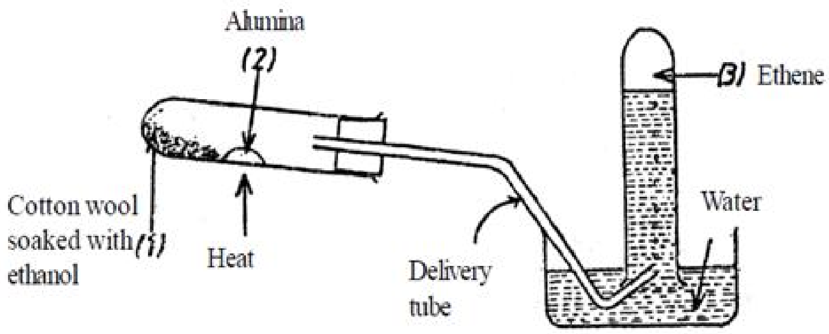


Figure 24.1: The apparatus used for the preparation of ethene

Procedure:

- Take ethanol to a height of about 2 cm to a boiling tube. With the help of a glass rod insert sufficient cotton wool to soak ethanol. Admit about 1 g of alumina and push it half - way down the tube.
- Fix the tube to a support and arrange the equipment as shown. Then heat the boiling tube directly under the location of the alumina. Collect the gas liberated into several boiling tubes by the downward displacement of water. They contain ethene. Perform following steps to the ethene gas produced.

(a) Add a few drops of bromine water.
(b) Add a few drops of alkaline potassium permanganate.
(c) Add a few drops of acidified potassium permanganate.
(d) Add ammoniacal silver nitrate solution.
(e) Add ammoniacal cuprous chloride solution.

Note:
Another way of doing above experiments is to take about 1 cm^3 of the reagent into a test tube and passing the gas into it.

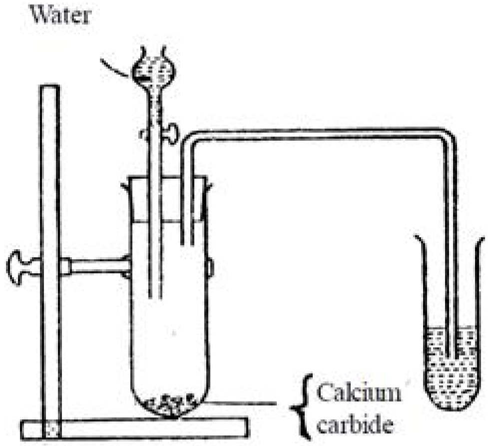


Figure 24.2: Apparatus for the preparation of ethyne

Take some calcium carbide into a boiling tube and arrange the apparatus as shown in Figure. Add few drops of water at a time.

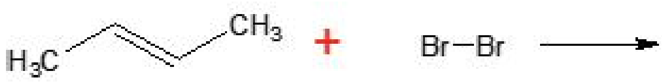
1. Take about 2.5 cm^3 of each of the following regents to a test tube and pass ethyne gas as shown in Figure.
   - Diluted bromine water
   - Alkaline potassium permanganate (very diluted)
   - Acidified potassium permanganate
   - Ammoniacal cuprous chloride
   - Ammoniacal silver nitrate

Note the observations. After the tests are carried out, wash the tubes thoroughly.

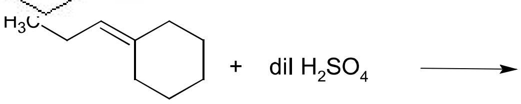
2. Hold a flame to the end of the delivery tube and allow the liberated ethyne to burn.

Post Lab questions:

Write down the major product of following reactions

1. 

2. 

3. 

4. Why it is necessary to use (very) diluted alkaline potassium permanganate for the test.

Experiment 25: Examination of the properties of alcohols.

Aim:

To observe reactions of the -OH group in alcohols.

Pre lab Questions:

1. What are the characteristics of alcohols?

Introduction:

Alcohols reacts with carboxylic acids to give esters, many of which have characteristic

odoure. Alcohols other than tertiary alcohols can be oxidized by various oxidizing agents.

Equipment an chemicals :

Equipment	Chemicals
Litmus paper Water bath Bunsen burner	Ethanol Methanol Isopropyl and butyl alcohols Acetic acid Sodium salicylate or salicylic acid Acidified potassium dichromate Acidified potassium permanganate Alkaline potassium permanganate Sodium carbonate

Experimental setup:

Take about 1 cm^3 of each of the given alcohols into three separate test tubes and do the following activities.

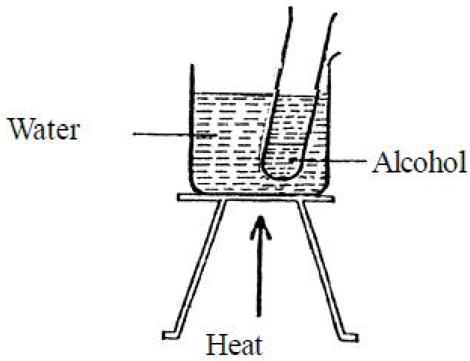


Figure 26.1: Experimental setup used for the alcohol testing

Precautions:

- Concentrated acids are very corrosive. Any spill should be immediately flushed with water. Dispose of reaction mixtures and excess reagents in proper containers as directed by your class teacher.

Procedure:

- Test the alcohol with blue and red litmus paper
- Add solid sodium carbonate and observe.
- Take about 1 cm^3 of the ethanol, isopropyl and butyl alcohols and add about 1cm^3 of glacial acetic acid. Add about 5 drops of conc. sulphuric acid and warm. Pour the product to cold water in a test tube and observe the smell. Add a few drops of bromine water.

OR

- Take about 1 cm^3 of the alcohol and add about 0.5 g of sodium salicylate or salicylic acid. Add about 5 drops of conc. sulphuric acid and warm. Pour the product into a test tube or a beaker containing cold water and observe the smell.

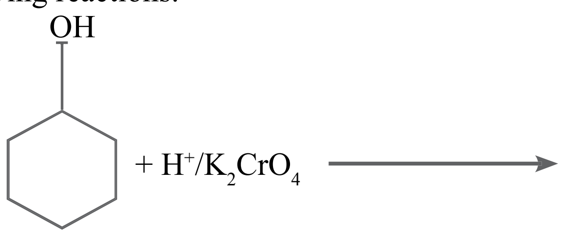
- Take 1cm^3 each of acidified potassium dichromate, acidified potassium permanganate and alkaline potassium permanganate to three test tubes separately. Add three drops of the alcohol to each. Repeat the same test with the other alcohols also. What colour changes do you observe in the solutions? How can you explain the colour changes? Examine the smell of the products.

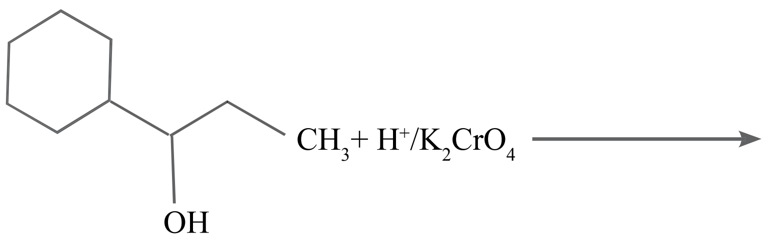
Note:

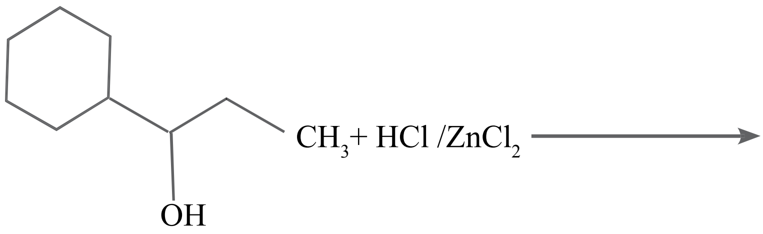
As the alcohols are inflammable, keep the bottles away from the burners.

Post Lab questions:

1. Write the structure of the major organic products expected from each of the following reactions.







2. What simple test can be used to distinguish between an alcohol and alkene?
3. What is the role of the concentrated H_2SO_4 acid in esterification reaction?

Experiment 26: Examination of the properties of phenol.

Aims:

1. To observe the acidic nature of phenol.
2. To observe the reactions of phenol.

Pre lab Questions:

1. Explain tests that can be used to differentiate an alcohol from a phenol
2. Explain why phenols are more acidic than alcohols?

Introduction:

Both alcohols and phenols contained an OH group. However, the properties of phenols are different from alcohols due to the fact that the OH group of phenol is attached to an aromatic ring.

Chemicals and items required:

Equipment	Chemicals
Litmus paper Water bath Bunsen burner	Phenol Sodium hydroxide solution Dilute hydrochloric acid Sodium carbonate solution Liquid bromine Dilute ammonia solution Dilute ferric chloride solution Alkaline potassium permanganate Sodium carbonate

Precautions:

- Concentrated solutions of the compound phenol are quite toxic and can cause severe skin burns. Phenol when comes into contact with the skin causes burns. Therefore it should be handled with care.

Procedure:

Take a little phenol separately into test tubes and do the following tests.

- Add 1 cm^3 of water and shake. Test the solution with red and blue litmus papers.
- Add 1 cm^3 liquid bromine.
- Add 1 cm^3 of sodium carbonate solution.
- Add few drops of neutral $FeCl_3$ solution and observe any colour change. To this solution add few drops of dil HCl and observe any colour change.

Note the observations.

***Preparation of neutral $FeCl_3$ solution:**

To a solution of dilute $FeCl_3$ solution add dil ammonium hydroxide until a faint permeant precipitation of ferric hydroxide is formed. Filter this solution to remove the brown ferric hydroxide precipitate to get the brownish yellow solution of neutral $FeCl_3$.

Results:

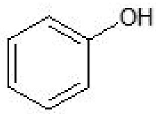
Record the observations of each test.

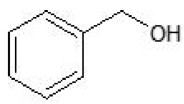
Discussion:

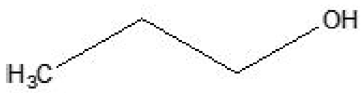
Using chemical reactions, explain the observations of each test performed for phenol.

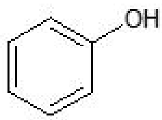
Post Lab questions:

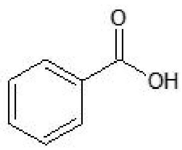
Explain how these compounds can be identified using chemical

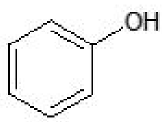
1. 

and 

2. 

and 

3. 

and 

Experiment 27: **Tests for aldehydes and ketones.**

Aim:

To observe some characteristic reactions of aldehydes and ketones.

Pre lab Questions:

1. Discuss the difference in the reactions of aldehydes and ketones with the following reagents.
   a. 2,4-DNP (water soluble) / Bready reagent (methanol)
   b. H^+ / $K_2Cr_2O_7$
   c. NH_3 / $AgNO_3$(aq)
2. Discuss the structural features of aldehydes and ketones which influence their solubility in water.

Introduction:

Aldehydes and ketones differ in their reaction because unlike in ketones aldehyde has at least one H atom attached to the carbonyl carbon. Aldehydes have at least one hydrogen attached to the carbonyl carbon; in ketones, no hydrogens are directly attached to the carbonyl carbon, only carbon containing alkyl (-R) groups.

Equipment and chemicals :

Equipment	Chemicals
Litmus paper Water bath Bunsen burner Test tubes	Methanal (Formaldehyde) Ethanal (Acetaldehyde) Benzaldehyde 2-propanone Acetophenone Acidified potassium permanganate Acidified potassium dichromate Ammoniacal silver nitrate solution (Tollen's reagent) 2,4 – dinitrophenylhydrazene (2,4-DNP) Fehling's solution (A and B)

Precautions: Aldehydes and ketones are toxic, therefore they should be handled with care.

Procedure:

- Solubility in water
- Do the following tests with benzaldehyde, propanone and acetophenon. Take about 1 cm^3 of water in a test tube and add the above compounds separately observing whether dissolve in water or they form two layers. (not more than 0.5 cm^3

- Reactions with 2,4-DNP
  - Dissolve acetaldehyde and acetone in water and add small amount of 2,4-DNP reagent and observe any change.
  - Dissolve a small amount of benzaldehyde and acetophenone separately in small amount of methanol and add small amount of Brady reagent.
- Take about 0.5 cm^3 of acetaldehyde, acetone and benzaldehyde dissolved in acetone in three different test tubes. Add to each test tube acidified $KMnO_4$ and observe any change that take place. Repeat the experiment with H^+ / $K_2Cr_2O_7$ in place of H^+ / $KMnO_{4.}$
- Prepare Fehling's solution by mixing 1cm^3 of Fehling A and 1cm^3 of Fehling B and add 1cm^3 these solutions and boil the mixture for 1 or 2 minutes. Repeat the same procedure to acetone in the place of acetaldehyde.
- Add 1-2 cm^3 of ammonical $AgNO_3$ in to a test tube containing 1 cm^3 of acetaldehyde and warm the solution in a water bath not more than 5 minutes.
- Repeat the same experiment with acetone in the place of acetaldehyde.
- Ammonical $AgNO_3$ (Tollens reagent) solution should be freshly prepared by the following procedure.
- Take about 1 cm^3 of $AgNO_3$ solution in a clean test tube and add drop of NaOH solution and add NH_4OH solution dropwise until the precipitate of Ag_2O is just dissolve.

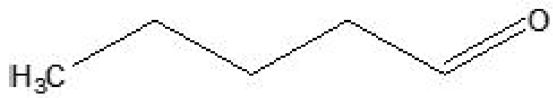
Note:
Use 2,4 - DNP reagent which is an aqueous solution for water-soluble carbonyl compounds. For carbonyl compounds that are insoluble in water, use brady reagent (2,4 - DNP in methanol).

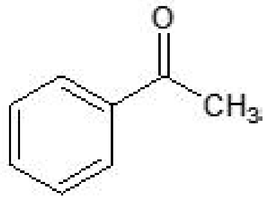
Results:
Write the observations for each test.

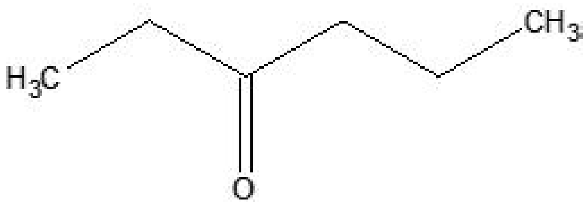
Discussion:
Explain the observations with reference to the reactions of each test performed for aldehyde and ketones.

Post Lab questions:

What are the observations for the following compounds with Tollens, Fehling, 2-4 DNP and Acidified $KMnO_4$.

1. 

2. 

3. 

Experiment 28: Examination of the properties of carboxylic acids.

Aim:

To observe the chemical properties of carboxylic acids.

Pre lab Questions:

1. Esters have various flavours and odours. List few of these and the alcohol and the carboxylic acid they are made of.
2. Write the structures of two water soluble carboxylic acids.
3. Write the main product of the reaction between benzoic acid and NaOH.
4. Name the naturally existing carboxylic acids in vinegar, lime juice and tomato.

Introduction:

Organic compounds with the carboxylic acid functional group are known as carboxylic acids. Carboxylic acids are found in many natural sources. Carboxylic acids give a sour taste to many foods. They are weak acids and dissociate partially in water.

Equipment and chemicals:

Equipment	Chemicals
Conical flask Test tubes Boiling tubes Beaker	conc H_2SO_4 Ammonical silver nitrate solution Acidified potassium permanganate solution Methanoic acid Ethanoic acid Benzoic acid Sodium carbonate Sodium hydroxide solution Sodium bicarbonate

Precautions: Sulfuric acid causes severe burns. Flush any spill with lots of water.

Procedure:

Do the following experiments using methanoic acid, ethanoic acid and benzoic acid. Tabulate the observations.

1. Mix with 1cm^3 of water and observe whether it is completely miscible.
2. Mix well with 2 cm^3 of dilute sodium hydroxide solution. Observe whether a clear solution is obtained.
3. Add to a solution of sodium carbonate and observe the evolution of gas bubbles.
4. Take about 1cm^3 of ethanol, add carboxylic acid and then a few drops of conc. sulphuric acid and heat gently for about one minute. Pour this mixture into an approximately 5% aqueous solution of $NaHCO_3$ in a beaker or watch glass and note the odour.

5. Add acidified potassium permanganate solution. Mix well and observe whether a colour change occurs.
6. Add about 2 cm^3 of ammonical silver nitrate (Tollens' reagent) and heat in a water bath. Observe whether a silver mirror is formed.

Results:

Acid	Test	Observation	Inference

Explain observations of each test conducted for carboxylic acids.

Post Lab questions:

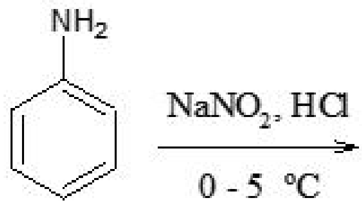
1. Discuss the reactions involved in the above tests.
2. How do carboxylic acids and esters differ in their chemical and physical properties?
3. Benzoic acid and diphenyl ketone are both solids which are insoluble in water. Suggest a method for separating a mixture of these compounds.

Experiment 29: Tests for aniline.

Aims:

1. To identify of aromatic primary amines by the chemical reactions of diazonium salts
2. To form azodyes via diazonium salts

Pre lab questions:



1. Draw the structure of the diazonium salt produced in the above reaction.
2. What are the possible by-products of the above reaction?
3. What are the product formed if the temperature is above 10 °C ?
4. Write the product of the reaction mentioned below.

$$CH_3CH_2NH_2 \xrightarrow[0\text{-}5\ °C]{NaNO_2\ /\ dil\ HCl}$$

Introduction:

Commercially available organic dyes are used in many industries such as food, clothing and paint. Azo dyes are one such commonly used class of synthetic dyes. All azo dyes have the basic structure of Ar-N=N-Ar', where Ar and Ar' are aromatic groups. In this laboratory experiment, aniline is converted to a diazonium salt which is then reacted with naphthalen-2-ol to produce an azo dye (2-napthol, β-naphthol).

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Ice bath	NaOH aniline 2-napthol or phenol $NaNO_2$ dil. HCl

Precautions: Aniline is a toxic substance. Avoid skin contact.

Procedure:

1. Dissolve about 0.2 g of 2-naphthol or phenol in 5 cm^3 of 10% NaOH solution and cool in an ice- bath.
2. Dissolve about 0.2 g of $NaNO_2$ in about 2 cm^3 of water and cool in an ice-bath.
3. Dissolve about 0.5 g of aniline in 2cm^3.
   A pale yellow solution obtained at the end of this step is the benzenediazonium salt solution.
   Add the benzendiazonium salt solution slowly to the previously prepared alkaline naphthalene-2-ol solution while keeping in the ice-water bath. Mixture must be stirred throughout this step. A bright orange-red precipitate is formed.
4. Mix about 0.5 g of aniline with 3 cm^3 of conc. HCl and dilute with 1 cm^3 of water and cool in an ice bath.
5. To this solution of aniline / HCl in the ice-bath, add slowly the cooled solution of $NaNO_2$, with stirring.
   [Note : If the addition is done too rapidly the heat of reaction can decompose the diazonium salt with the evolution of bubbles of N_2 gas.]

Discussion:

Explain observations of each step producing a colour change.

Post lab questions:

1. What is the coloured product? Write the chemical equation for the formation of this product.
2. Can similar dyes be produced using aliphatic amines? Explain your answer.
3. Explain why the dye formation reaction is carried out in an alkaline medium.

Experiment 30 : Experimental determination of the effect of acid concentration on the reaction rate between Mg and acid reaction

Aims:

1. To investigate the effect of the concentration of reactants on the rate of a reaction
2. To determine the order of the reaction between Mg and HCl acid by measuring the volume of H_2 gas evolved

Pre lab questions

1. Draw the conventional pattern of the graph for the decreasing concentration of a reactant participating in a reaction with time. Indicate the initial rate, instantaneous rate and average rate in the graph.
2. Draw the conventional pattern of the graphs of concentration versus time for a simple reaction A → B, when the reaction is (i) *zero order* (ii) *first order and* (iii) *second order.*
3. A 100.0 g sample of $CaCO_3$ was placed in a beaker and 100 cm^3 of 1.0 mol dm^{-3} HCl acid was added. After 30 s, the weight of remaining $CaCO_3$ was found to be 70.0 g.
   (i) Calculate the average rate of the reaction.
   (ii) If the reaction consumes $CaCO_3$ at an average rate of 0.20 mol s^{-1} during the first 10 s, what mass of $CaCO_3$ remains unreacted?

Introduction

The study of the rates of chemical reactions and the ability to control them are the targets of learning chemical kinetics. The factors such as concentration, temperature, catalysts, physical nature of the reactants affect the rate of reactions and the mechanisms by which reactions proceed. If the rate of a chemical reaction is fast, a large portion of the molecules react to form products in a given time period. If the rate of a chemical reaction is slow, a small portion of molecules react to form products in a given time period. In general, the rate of a reaction indicates how fast the consumption of reactants or the formation of products occurs. For a simple reaction of A → P, we can write the rate as $-\frac{\Delta C_A}{\Delta t}$, or $\frac{\Delta C_P}{\Delta t}$ i.e. the ratio of the change in concentration in a known time interval. However, there are instances where the rate is defined in different ways; in a known time interval as the one described above, at the starting point of the reaction, at a point of given time. Therefore, it is useful to understand those basic definitions together with the idea how the rate of reaction varies over the time. These can be better understood by simple experiments and by graphical representation of the experimental data.

As we know, the rate of a reaction has direct relationship with the concentration of reactants and for a reaction, $A + B \rightarrow C + D$, the rate is expressed as:
Rate α $[A]^n [B]^m$ or Rate = $k [A]^n [B]^m$

It is referred to as the rate law or the rate expression of the reaction and ***n*** and ***m*** are called the order with respect to reactants A and B, respectively.

In this experiment, we will understand how to design an experiment to measure the variation of reaction rate with the concentration of HCl and also to estimate the order of reaction with respect to HCl. For the reaction:
$Mg(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2(g)$ the rate expression can be written as follows.

$$\text{Rate} = k\,[Mg(s)]^n\,[HCl(aq)]^m$$

[Mg(s)] is a constant due to a solid.

$$\text{Rate}\ \alpha\ [HCl(aq)]^m$$

As H_2 gas evolves, the kinetics of the reaction can be followed by measuring the time taken to produce a constant volume of H_2 gas by changing the concentration of HCl solution.

$$\text{Rate}\ \alpha\ [HCl(aq)]^m$$

$$\text{Rate}\ \alpha\ [\text{Volume of } H_2 / \text{Time (t)}]^m$$

As the volume of H_2 is a constant,

$$\text{Rate}\ \alpha\ (1/t)^m$$

Equipment and chemicals required:

Equipment	Chemicals
Boiling tube Rubber stoppers fitting to boiling tubes Glass tubes Stop watch Beakers	500 cm^3 1.0 mol dm^{-3} HCl acid Cleaned Mg strips of 3 cm in length

Procedure:

1. Make a mark on the boiling tube with a pen about 3 cm above from the bottom of the boiling tube.
2. Connect a glass tube to a cork and also fix a Mg strip to the end of the glass tube as shown in Figure 31.1. (It is necessary to keep the same length of the Mg strip exposed to react in each experiment.)
3. Fill the boiling tube completely with 1. 0 mol dm^{-3} HCl acid and close it with the rubber stoppers attached with a glass tube and a Mg strip.
4. Turn the boiling tube upside down and start the stop watch immediately.
5. Record the time taken for the liquid level to reach the mark on the boiling tube.

6. Repeat the steps 3 to 5 for new Mg strips with different concentrations of HCl acid (for example: 0.80, 0.60, 0.40 and 0.20 mol dm^{-3})
   (Note:The concentrations of HCl solutions can be adjusted by selecting appropriate volumes of 1.00 mol dm^{-3} HCl and the volume of the distilled water by taking the total volume of the test tube.)
   Tabulate your data of time taken to collect the constant volume of H_2 gas for different concentrations of HCl and calculate rate at each concentration.

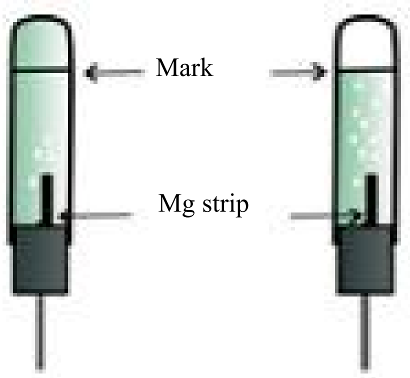


Figure 31.1 Experimental set-up

[HCl]/mol dm^{-3}	Time/s	Rate/mol d m^{-3} s^{-1}
1.0		
0.8		
0.6		
0.4		
0.2		

Determine the order of the reaction with respect to [HCl(aq)] using above data.
Draw a suitable graph for the above data and comment on that.

An alternative experimental set-up

The following arrangement can also be used for the above experiment.

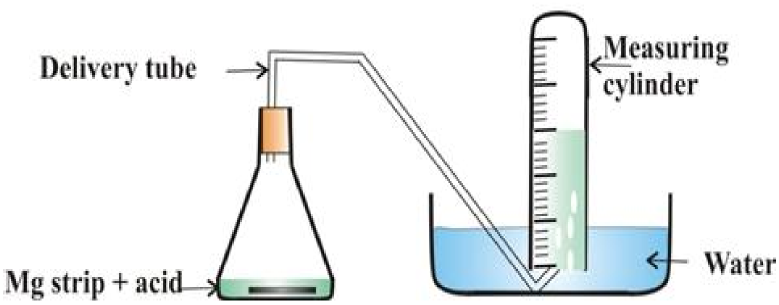


Figure 31.2 Experimental set-up

Additional experiment: Experimental determination of the order of the reaction between $CaCO_3$(s) and hydrochloric acid

Aim:

To determine the order of reaction between $CaCO_3$(s) and hydrochloric acid by measuring the mass loss.

Introduction:

When reacted with HCl acid, $CaCO_3$ produces $CaCl_2$, CO_2 and H_2O.

$$CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g)$$

In this experiment the rate is determined by measuring the weight loss of $CaCO_3$.

Equipment and chemicals required:

Equipment	Chemicals
Beakers Electronic balance Stop-watch	300 g of $CaCO_3$ (s) 300 cm^3 each of 1.0 mol dm^{-3}, 1.5 mol dm^{-3}, and 2.0 mol dm^{-3} HCl solutions (*these can be made by dilution*).

Procedure:

1. Add about 100 g of $CaCO_3$(s) into a beaker and measure the total weight (weight of $CaCO_3$ + weight of an empty beaker).
2. Add 100 cm^3 of 1.0 mol dm^{-3} HCl solution and record the weight every 20 seconds by keeping the beaker with the mixture on the electronic balance.
3. Repeat steps 1 and 2 with 1.5 mol dm^{-3} and 2.0 mol dm^{-3} HCl solutions.

Record your results in the following table:

Time/s	Weight loss /g		
	$CaCO_3$(s) with 1.0 mol dm^{-3} HCl	$CaCO_3$(s) with 1.5 mol dm^{-3} HCl	$CaCO_3$(s) with 2.0 mol dm^{-3} HCl

Using data,

1. plot the graph of weight loss against time for the reaction between $CaCO_3$ (s) and 1.0 mol dm^{-3} HCl. Calculate the average rate in 100 to 200 second interval.

2. plot the graph of weight loss against time for each experiment in one graph and comment on your observations.
3. calculate the reaction rates in 20 second time intervals
for example: (*weight at 40 s – weight at 20 s)g / 20 s = x g* s^{-1}
4. draw the graphs of rate against the concentration of HCl.
5. calculate the order of the reaction with respect to HCl.

Post lab questions

1. Is it possible to find the order of the reaction with respect to HCl in the above experiment between $CaCO_3$ and hydrochloric acid? Comment.
2. If 2.0 g weight loss occurred during first 50 seconds of the reaction between powdered $CaCO_3$(s) with 2.0 mol dm^{-3} HCl, calculate the concentration of HCl at 50 s.
3. Following table shows the data obtained at room temperature for the reaction:
$NO_2(g) + CO(g) \rightarrow NO(g) + CO_2(g)$
Using the data, determine the order of reaction with respect to each reactant and overall rate law for this reaction. Comment on your findings.

Experiment	[CO]/ mol dm^{-3}	[NO_2]/mol dm^{-3}	Initial rate/ mol dm^{-3} s^{-1}
1	0.1	0.1	0.005
2	0.1	0.4	0.080
3	0.2	0.1	0.005

Experiment 31: Experimental determination of the effect of concentration on the rate of the reaction between $Na_2S_2O_3$ and HNO_3.

Aims:

1. To investigate the effect of concentration of the reactants on the rate of a reaction
2. To determine the order of the reaction and write the rate equation for the reaction.

Pre lab questions

1. Write the balanced chemical equation for the reaction between $Na_2S_2O_3$ and HNO_3.
2. What are the observations of the above reaction ?

Introduction

In this experiment the reaction between $Na_2S_2O_3$ and HNO_3 is used.

$$Na_2S_2O_3(aq) + 2HNO_3(aq) \rightarrow S(s) + 2NaNO_3(aq) + SO_2(g) + H_2O(l)$$

To investigate the aims mentioned above, it is easy to measure a time taken for the formation of a constant amount of solid sulfur as a product of the reaction. Therefore, the rate of formation of solids sulfur can be considered the rate of the above reaction. The results will be analyzed to determine the order of the reaction and hence the rate expression of the reaction can be derived.

Equipment and chemicals required:

Equipment	Chemicals
Beakers White paper with a cross mark in black Stop-watch	3.0 mol dm^{-3} HNO_3 acid solution 40 g dm^{-3} (or 0.15 mol dm^{-3}) sodium thiosulfate solution

Procedure

Put the beaker on the piece of paper with "X" mark. Allow the reaction to start by adding the volume of $Na_2S_2O_3$ into the beaker and then the volume of HNO_3 according to the given table 31.1 and table 31.2. It is important to start the stop watch at the point of addition of acid in each experiment. Measure the time taken to disappear mark "X". Due to the formation of sulfur, colour changes from colourless to cloudy yellowish.

Table 31.1 Determination of the rate by varying the volume of $Na_2S_2O_3$

Experiment	Volume of 0.15 mol dm^{-3} $Na_2S_2O_3$/ cm^3	Volume of 3.0 mol dm^{-3} HNO_3/ cm^3	Volume of distilled water/ cm^3	Time taken for disappearance of "X"	Rate
1	25.0	5.0	0.0		
2	20.0	5.0	5.0		
3	15.0	5.0	10.0		
4	10.0	5.0	15.0		
5	5.0	5.0	20.0		

Table 31.2 Determination of the rate by varying the volume of HNO_3

Experiment	Volume of 0.15 mol dm^{-3} $Na_2S_2O_3$/ cm^3	Volume of 3.0 mol dm^{-3} HNO_3/ cm^3	Volume of distilled water/ cm^3	Time taken for disappearance of "X"	Rate
1	25.0	5.0	0.0		
2	25.0	4.0	1.0		
3	25.0	3.0	2.0		
4	25.0	2.0	3.0		
5	25.0	1.0	4.0		

Note; *It is necessary to observe the mark "X" from a fixed height of the beaker.*

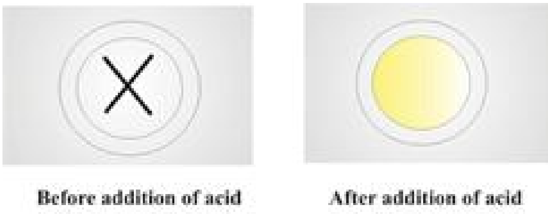
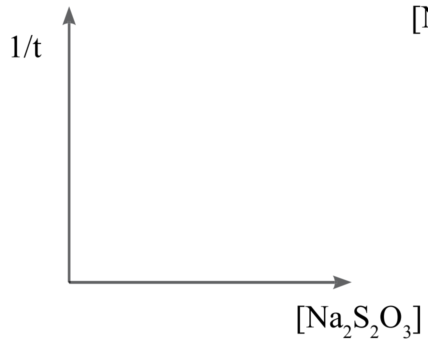


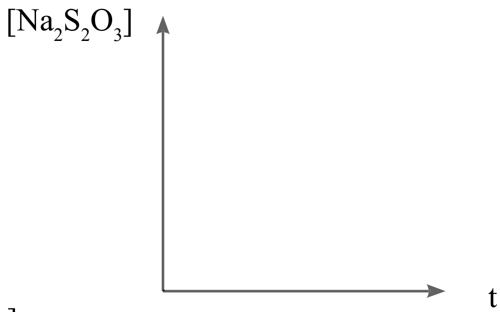
Figure 31.1

Determine the order of the reaction with respect to $[HNO_3(aq)]$ and $[Na_2S_2O_3(aq)]$ using above data and write the rate expression.
Draw a suitable graph using the above data and comment on it.

Post lab questions

1. What are the reasons for keeping HNO_3 concentration at a higher level with respect to the $Na_2S_2O_3$ concentration?
2. Draw the following graphs.





Experiment 32 : Experimental determination of the order of reaction with respect to Fe^{3+} for the reaction between Fe^{3+} and I^-

Aim:

To determine the order and the rate expression of a reaction experimentally

Pre lab questions

1. If the rate law for a reaction is $R = k[A]^2[B]$,
   - (a) What is the overall order of the reaction?
   - (b) If the concentration of both A and B are doubled, how will this affect the rate of the reaction?
   - (c) When the concentration of A is doubled, while the concentration of B is kept constant, how is the value of k (rate constant) affected at a constant temperature?

Introduction

In this experiment, the kinetics of the following reaction is investigated.

$$2I^-(aq) + 2Fe^{3+}(aq) \rightarrow I_2(aq) + 2Fe^{2+}(aq)$$

The experimental procedure given below should be followed, in order to determine the values of *a, b and k* in the following rate expression .

$$Rate = k\,[Fe^{3+}(aq)]^a\,[I^-(aq)]^b$$

Changing the concentration of the ***Fe³⁺*** is not an easy task in the above reaction. However, we can think about some appropriate method: As we can see that iodine, $I_2(aq)$ is produced in the reaction which could be allowed to react with a second reactant which has no effect on either Fe^{3+} or I^-, thiosulfate, $S_2O_3^{2-}(aq)$ can be used as a second reactant, which reacts with $I_2(aq)$ as follows.

$$I_2(aq) + 2\,S_2O_3^{2-}(aq) \rightarrow 2\,I^-(aq) + S_4O_6^{2-}(aq)$$

This reaction will occur simultaneously with the reaction under investigation. Adding starch to the mixture allows the $S_2O_3^{2-}(aq)$ of the second reaction to act as a built in "clock" (indicator) as the mixture turns blue when all the $S_2O_3^{2-}$ (aq) is consumed. As soon as the $S_2O_3^{2-}$ (aq) is consumed, any additional $I_2(aq)$ formed by the reaction between ferric and iodide ions will react with the starch to form a complex with the characteristic blue colour. Thus, a known fixed amount of $S_2O_3^{2-}$ (aq) with starch is added to the reaction mixture and allowed to react with the $I_2(aq)$ produced.

Therefore, the following reactions occur simultaneously in the mixture.

$2I^-(aq) + 2Fe^{3+}(aq) \rightarrow I_2(aq) + 2Fe^{2+}(aq)$ *slow*

$I_2(aq) + 2\,S_2O_3^{2-}(aq) \rightarrow 2\,I^-(aq) + S_4O_6^{2-}(aq)$ *fast*

At the instance of the appearance of blue colour, the decrease in the concentration of Fe^{3+} from its initial value is just equal to the initial concentration of $S_2O_3^{2-}$ (aq) in the mixture. Thus the initial rate;

$$-\frac{1}{2}\frac{d\left[Fe^{3+}(aq)\right]}{dt} = \frac{1}{2}\frac{d\left[S_2O_3^{2-}(aq)\right]_i}{dt} \approx \frac{1}{2}\frac{d\left[S_2O_3^{2-}(aq)\right]_i}{t}$$

$\left[S_2O_3^{2-}(aq)\right]i$ is the initial (fixed) concentration of $\boldsymbol{S_2O_3^{2-}(aq)}$ and dt ≈ t is the time in seconds taken for blue colour. The above relation is valid if the initial concentration of thiosulfate is sufficiently low compared to that of Fe^{3+}(aq) and I^-(aq) ions.

First, the evaluation of exponents ***a*** and ***b*** is experimentally carried out and then the calculation of the rate constant k with the help of initial rate method.

Equipment and chemicals :

Equipment and Items	Chemicals
Beakers Measuring cylinders Stop-watch	Acidified 0.10 mol dm^{-3} and 1 mol dm^{-3} ammonium iron (III) sulfate solution 0.2 mol dm^{-3} and 3.0 mol dm^{-3} potassium iodide solutions 0.006 mol dm^{-3} sodium thiosulfate solution Starch solution Distilled water

Procedure

Note: *Acidified ammoniumiron(III) solution is prepared by dissolving the required weight of reagent in 1. 00 mol dm^{-3} H_2SO_4*

Starch solution is prepared by dissolving about 5 g of starch in 100 cm^3 of hot water.

The reaction is started by mixing a colourless acidic solution of Fe^{3+}(aq) with a colourless solution containing sodium thiosulfate, potassium iodide and about 5 drops of starch solution.

A. Reaction order with respect to Fe^{3+}(aq)

1. Prepare the mixtures for experiment A by adding the solutions specified in Table 1 to 100 cm^3 beakers, respectively.

Beaker I: Fe^{3+}(aq) solution, Dil. H_2SO_4
Beaker II: I^-(aq) + $S_2O_3^{2-}$(aq) + starch + Water

Experiment	Dil Water/cm^3	Acidified 0.10 mol dm^{-3} Fe^{3+}(aq)/ cm^3	3.0 mol dm^{-3} KI(aq)/ cm^3	0.006 mol dm^{-3} $S_2O_3^{2-}$ (aq) + starch/ cm^3	Time taken for blue colour to appear (t)
1	0.0	25.0	10.0	15.0	
2	5.0	20.0	10.0	15.0	
3	10.0	15.0	10.0	15.0	
4	15.0	10.0	10.0	15.0	
5	20.0	5.0	10.0	15.0	

Add the contents in the beaker I to the beaker II start the stop watch simultaneously and swirl the solutions until mixed. Stop the timer at the first appearance of the blue colour. Record the time, t. *Repeat these steps for all 5 experiments.*

B. Reaction order with respect to I^-(aq)

This part is performed exactly as in Part A, except that the concentration of ferric ion is kept constant while the concentration of iodide ion is varied as given in the table below.

Experiment	Dil Water/cm^3	Acidified 0.10 mol dm^{-3} Fe^{3+}(aq)/ cm^3	3.0 mol dm^{-3} KI(aq)/ cm^3	0.006 mol dm^{-3} $S_2O_3^{2-}$ (aq) + starch/ cm^3	Time taken for blue colour to appear (t)
1	0.0	25.0	10.0	15.0	
2	2.0	25.0	8.0	15.0	
3	4.0	25.0	6.0	15.0	
4	6.0	25.0	4.0	15.0	
5	8.0	25.0	2.0	15.0	

Calculations: .

1. Calculate [$S_2O_3^{2-}$*(aq)*], which is constant in all experiments.
2. Calculate [Fe^{3+}(aq)] and [I^-(aq)] separately at different time intervals found in tables of A and B.
3. Calculate initial reaction rate, by using time data above.
   It will be noted that the calculated rate can easily be obtained by the value of 1/t. (This can be confirmed by estimating the percent change in the concentration of $S_2O_3^{2-}$*(aq)*. As it is very small, we can assume that it is a constant).
4. Plot the graphs $\frac{1}{t}$ *vs [Fe^{3+}(aq)]* and $\frac{1}{t}$ vs [I^-(aq)] separately and comment on the shapes of the graphs.
5. Determine the values of *a, b and k* using the data obtained and hence the rate expression of the reaction.

Note: It will be noted that the calculated initial rate values are directly proportional to the reciprocal of time measured and hence we can use $\frac{1}{t}$ values as reaction rates. In addition, as the total volume of the reaction mixture is constant throughout the experiments, concentrations of the respective species will then be directly proportional to their volumes (V) used in each experiment. Therefore, from the plots of $\frac{1}{t}$ vs V, the orders of the recation with respect to each reactant can easily be obtained and understood.

Post lab questions

1. Write the chemical equation for the iodide-catalyzed decomposition of H_2O_2.
2. It is found that the doubling of the concentration of either A or B quadruples the reaction rate of a unimolecular reaction; $2A + B \rightarrow C$. Write a rate law for the reaction.
3. When the concentration of a substance is doubled, what effect does it have on the rate if the order with respect to that reactant is (a) 0 (b) 1 and (c) 2
4. Can you add Fe^{3+} and $S_2O_3^{2-}$ together? Give reasons.
5. Can you add H_2SO_4 and $S_2O_3^{2-}$ together? Give reasons.
6. What is the reason for keeping KI concentration in a higher level than other solutions in experiment A?
7. Why water is added to the mixture?

Experiment 33: Experimental study of the characteristics of a dynamic equilibrium system using Fe^{3+} and SCN^- system

Aims:

1. To develop a better understanding of a dynamic chemical equilibrium
2. To investigate the effect of the application of some stress on the equilibrium position of an equilibrium system.

Pre lab questions:

Iron(III) has a coordination number of 6 and aqueous solutions of iron(III) salts are generally yellow in colour due to the presence of hydroxo-complexes of the iron(III) cation. They are converted into brown aqua-complexes in the presence of an acid.

1. Write the balanced equilibrium reaction for the above system.
2. Write the balanced equation for the reaction between the aqueous complex and aqueous SCN^- ions.

Introduction:

It is possible to shift the equilibrium in a desired direction by applying a stress to the system. This process is explained by Le Chatelier's Principle, which states that, "When a system at equilibrium is subjected to a stress, the system will react so as to relieve the stress." Some examples of stresses that can be applied to an equilibrium system are changes in concentration (both increasing and decreasing), pressure (for systems involving gases), and temperature.

Note: Changing concentration of reactants and/or products will never change the equilibrium constant of a reaction. In this experiment the effects of change in concentration will be observed.

Equilibrium constant for the reaction; $Fe^{3+}(aq) + SCN^-(aq) \rightleftharpoons [FeSCN]^{2+}(aq)$ is given below.

$$K_C = \frac{\left[[FeSCN]^{2+}(aq)\right]}{\left[Fe^{3+}(aq)\right]\left[SCN^-(aq)\right]}$$

A standard laboratory example for demonstrating the effect of changing concentrations on the equilibrium position of the reaction $Fe^{3+}(aq) + SCN^-(aq) \rightleftharpoons [FeSCN]^{2+}(aq)$ can be followed by observing the colour of the system upon the addition of an appropriate reagent to the equilibrium mixture.

$$Fe^{3+}(aq) + SCN^-(aq) \rightleftharpoons [FeSCN]^{2+}(aq)$$

pale yellow *blood red*

The original colour of the above equilibrium system would be orange (it is a mixture of pale yellow and red). The position of the above equilibrium can be determined from the colour of the solution. The original colour of the mixture is affected when an appropriate reagent is added, which causes a shift in the equilibrium position. If the equilibrium lies to the right, the solution is blood red in colour and if the equilibrium lies to the left, the solution is pale yellow in colour. This effect can be studied by a simple experiment described below.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes	100 cm^3 of 0.05 mol dm^{-3} iron(III) nitrate ($Fe(NO_3)_3$) or $FeCl_3$ in 0.5 mol dm^{-3} HNO_3 100 cm^3 of 0.005 mol dm^{-3} potassium thiocyanate (KSCN) or sodium thiocyanate or ammonium thiosyanate Dilute (1.0 mol dm^{-3}) sodium hydroxide (NaOH) Solid $Fe(NO_3)_3$ or $FeCl_3$ and KSCN

Procedure:

- Add about 2 cm^3 of iron(III) solution and 2 cm^3 of thiocyanate solution in to a test tube and mix well. Dilute appropriately to get a pale orange coloured solution.
- Divide the prepared solution into four test tubes.
- Keep one test tube with the solution without altering and use it as the control.
- To the second test tube, add 10 drops of sodium hydroxide. Record your observations.
- To the third test tube, add a little amount of solid $Fe(NO_3)_3$ and dissolve it (or add 10 drops of iron(III) nitrate). Record your observations.
- Add a little amount of solid KSCN (or 10 drops of potassium thiocyanate) to the fourth tube and record your observations.

Observations:

Record your observations as follows.

Tube	Colour after adding NaOH	Colour after adding Fe^{3+}	Colour after adding SCN^-
1 (control)	orange	orange	orange
2			
3			
4			

Post lab questions:

1. Use Le Châtelier's principle to explain the above observations.
2. Why is an acidic medium necessary for the above studied system?
3. In an experiment 10.0 cm^3 of 2.0×10^{-3} mol dm^{-3} $Fe(NO_3)_3$ and 10.0 cm^3 of 2.0×10^{-3} mol dm^{-3} KSCN solutions were mixed. After the system attained the equilibrium the concentration of $[FeSCN]^{2+}$ was found to be 1.5×10^{-4} mol dm^{-3} at room temperature. Calculate the equilibrium constant of the reaction.

Experiment 34: Experimental study of the effect of temperature on the system of $NO_2(g)$ and $N_2O_4(g)$

Aims:

1. To develop a better understanding of dynamic chemical equilibrium in a gaseous system
2. To investigate the effect of the application of some stress on the equilibrium position of a equilibrium system.

Pre-lab questions:

What are the methods of preparing NO_2 gas in the laboratory?
Write the balanced chemical equations for the above chemical reactions.
What is the colour of a sample of NO_2 gas?

Introduction:

As we know, changes in concentration, temperature or pressure can stress an equilibrium system and may cause a shift in the position of equilibrium. In this demonstration, the effect of temperature on the following equilibrium system will be investigated. If a sample of NO_2 gas is taken, it exists as a mixture of NO_2 and N_2O_4.

$$2NO_2(g) \rightleftharpoons N_2O_4(g) \qquad \Delta H = -58\ kJ\ mol^{-1}$$

brown *colourless*

This reaction is exothermic in the forward reaction (produces N_2O_4), and endothermic in the reverse reaction (produces NO_2). This is becauseN_2O_4 is more stable than NO_2 (the bonds in N_2O_4 are stronger than the bonds in NO_2).
An increase in temperature will cause the reaction to shift in a way that uses the excess heat (favours the endothermic reaction), while a decrease in temperature will result in a shift to produce heat (favours the exothermic reaction).
The following experimental procedure will enable us to test a system of this nature.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Rubber corks Delivery tube with a cork Beakers	Conc. HNO_3 acid Ice Hot water Cu turnings

Procedure:

- Add about 5 g of Cu turnings into a boiling tube and add about 5 cm^3 of conc. HNO_3 acid.
- Connect the delivery tube immediately and collect the evolved gas into three identical test tubes so that the glass tubes get the same colour intensity.
- Close the test tubes tightly with rubber corks.
- Place NO_2/N_2O_4 tubes one each in beakers containing cold water, water at room temperature and warm water respectively.
- Observe the relative variation in the colour intensity of them (compared to the one at room temperature).
- Determine the direction of shift in the equilibrium position of the reaction.

$$\mathbf{2NO_2(g) \rightleftharpoons N_2O_4\ (g)}$$

- Interpret your observations appropriately for the three temperatures.

Post lab questions:

1. Explain using suitable graphs the variation in concentrations of NO_2(g) and N_2O_4(g) in the $2NO_2(g) \rightleftharpoons N_2O_4(g)$ equilibrium system when the following instantaneous changes are applied. Graphically show how the changes applied affect the forward and backward reactions until the system re-establishes the equilibrium.
   a. More NO_2(g) is added.
   b. NO_2(g) is removed.
   c. More N_2O_4(g) is added.
   d. N_2O_4(g) is removed.
   e. Temperature is increased.
   f. Temperature is decreased.

2. Explain the observations that you expect in increasing pressure of the above system.

Additional Experiment: Experimental study of the effect of temperature on the system of $Co(H_2O)_6^{2+}(aq)/CoCl_4^{2-}(aq)$

Aim:

To facilitate further understanding of Le Châtelier's principle by referring to the equilibrium system;

$$Co(H_2O)_6^{2+}(aq) + 4Cl^-(aq) \rightleftharpoons CoCl_4^{2-}(aq) + 6H_2O(l)$$

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Boiling tubes Beakers (250 cm^3) Eye dropper pipette	Solid cobalt(II) chloride ($CoCl_2.6H_2O$) Solid calcium chloride ($CaCl_2$) 0.1 mol dm^{-3} silver nitrate ($AgNO_3$) 12 mol dm^{-3} hydrochloric acid (HCl) Ethanol Ice, hot water

Procedure:

- Measure out 25 cm^3 of ethanol and place it in a 50cm^3 beaker.
- Add 4 or 5 crystals of the cobalt(II) chloride to the ethanol in the beaker until a blue solution results. Add more crystals, if necessary.
- Transfer 5 cm^3 each of the above solution into four separate test tubes using an eyedropper pipette.
- To one of the tube from step 3, add distilled water drop-wise and record your observations after each drop. Repeat this step in two more tubes so that all three of them exhibit the same colour.
- Add concentrated hydrochloric acid to one of the tubes in step 4 drop-wise and record your observations.
- To the second tube from step 4, add a small amount of solid calcium chloride.
- To the third tube from step 4, add 10 drops of 0.1 mol dm^{-3} silver nitrate.
- Use the fourth as a control.
- To the remaining solution in the beaker, add just enough distilled water to get a purple color that is about halfway between the blue and pink shades. Warm the beaker until a colour change occurs.
- Cool the beaker with the mixture in 9 above in an ice bath and observe and record the colour change.

List the observations in following table.

Test (Addition of)	Colour	
	Before	After
Distilled water		
Solid $CoCl_2 . 6H_2O$		
$CaCl_2$		
HCl		
$AgNO_3$		
Heating		
Cooling		

Use Le Châtelier's principle to explain the observations in each steps of the procedure.

Post lab questions:

1. Which cobalt complexes were favoured by the addition of each reagent in the above procedure? Explain your answers with the help of Le Châtelier's principle.

2. Which cobalt complex was favoured by the addition of heat in step 9 of the procedure? Rewrite the equation for the reaction, including the energy term directly in the equation. The value of ΔH for the process is + 50 kJ mol^{-1}. Use Le Châtelier's principle and the equation that you just wrote to explain the colour changes that resulted from heating and cooling.

Experiment 35 : Experimental determination of the acidic/basic/neutral nature of aqueous solutions of salts by testing pH

Aim :

To classify given salts as acidic, basic or neutral by referring to the pH value of their aqueous solutions.

Pre lab questions:

1. Name three salts that are used in day to day life.
2. State by giving balanced chemical equations how the following salts are formed by the neutralization reactions between acids and bases.

   $NaCl$, $(NH_4)_2SO_4$, CH_3COONa

Introduction:

Salts are derived from the neutralization reactions between acids and bases. Salts are solid crystalline ionic compounds that are composed of cations and anions. They have high melting points. Aqueous solutions or molten liquids of the salts are strong electrolytes. Based on the way that constituent ions of a salt undergo hydrolysis, salts can be grouped under three categories.

Equipment and chemicals required:

Equipment	Chemicals
Test tubes Measuring cylinder (10 cm^3) Balance White paper	sodium chloride sodium acetate ammonium acetate ammonium chloride zinc chloride aluminium chloride magnesium sulphate any other suitable salts universal indicator or pH papers distilled water

Method:

- Weigh approximately 0.5 g of each salt and put them into cleaned test tubes separately.
- Prepare aqueous solutions of each salt by dissolving in 10 cm^3 of distilled water.
- Add 10 cm^3 of water to a separate test tube.
- Add 2 drops of universal indicator or a piece of pH paper into each test tube.
- Keep a white paper behind the test tubes in order to observe colours of the solutions clearly.
- Determine the approximate pH value of each solution by comparing with the relevant colour code.

- Observe that the dissolving of some salts changes the pH of distilled water by referring to the test tube containing the sample of distilled water.
- Complete the following table.

Salt solution	NaCl	CH_3COONa	CH_3COONH_4	NH_4Cl	$ZnCl_2$	$AlCl_3$	$MgSO_4$	water
Rough pH value								

a. Classify the salts used as acidic, basic or neutral based on the rough pH values of their aqueous solutions.
b. Predict the acidic, basic or neutral nature of the salt based on the acid and the base by which the salt is formed.

Post lab questions:

1. Give examples for the acidic, basic and neutral salts which are available in the laboratory.
2. Write a relationship between the nature of the acid and base participated in formation of the salt and the acidic/basic/neutral nature of the salt.
3. Classify the following salts as acidic, basic or neutral.
   C_6H_5COONa, CH_3COOK, Na_3PO_4, $BaSO_4$, KBr, Na_2CO_3, $NaNO_3$, $(NH_4)_2SO_4$

Experiment 36: Titration between Na_2CO_3 and HCl using phenolphthalein and methyl orange

Aims:

1. To learn the shape of the titration curve for the titration between a solution of Na_2CO_3 which acts as a diprotic base and HCl
2. To investigate the pH ranges of the equivalence points and detecting them with suitable indicators

Introduction:

Carbonate ion, $CO_3^{2-}(aq)$, is a diprotic base, and ionizes in two steps as follows in aqueous solution. For each of the following equilibrium we can define base ionization constants K_{b_1} and K_{b_2}

$$CO_3^{2-}(aq) + H_2O(l) \rightleftharpoons HCO_3^-(aq) + OH^-(aq) \qquad K_{b_1} = \frac{[HCO^-_{3(aq)}][OH^-_{(aq)}]}{[CO^{2-}_{3(aq)}]}$$

$$HCO_3^-(aq) + H2O(l) \rightleftharpoons H_2CO_3(aq) + OH^-(aq) \qquad K_{b_2} = \frac{[H_2CO_{3(aq)}][OH^-_{(aq)}]}{[HCO^-_{3(aq)}]}$$

Therefore, when Na_2CO_3 reacts with HCl, the neutralization occurs in two consecutive steps. At the beginning of the titration, $H^+(aq)$ ions from HCl(aq) react with $CO_3^{2-}(aq)$ ions as carbonate ion is the strongest base present in the initial mixture.

$$H^+(aq) + CO_3^{2-}(aq) \rightarrow HCO_3^-(aq)$$

Then, in the second reaction, protons from HCl(aq) react with the hydrogencarbonate ions formed in the first reaction.

$$H^+(aq) + HCO_3^-(aq) \rightarrow H_2CO_3(aq)$$

Two equivalence points can be detected, one when all of the $CO_3^{2-}(aq)$ has been converted to the amphiprotic $HCO_3^-(aq)$, and second when all of the $HCO_3^-(aq)$ has subsequently been converted to $H_2CO_3(aq)$.

It is clear that at the first equivalence point the following equilibrium exists in the mixture and therefore the pH is determined by the hydrolysis of $HCO_3^-(aq)$ ions.

$$HCO_3^-(aq) + H_2O(l) \rightleftharpoons H_2CO_3(aq) + OH^-(aq)$$

At the second equivalence point the following equilibrium exists in the mixture and therefore the pH is determined by the hydrolysis (first dissociation) of H_2CO_3 (aq) acid.

$$H_2CO_3\,(aq) + H_2O(l) \rightleftharpoons HCO_3^-(aq) + H_3O^+(aq)$$

By looking at the above two reactions one may note that, at the first equivalence point mixture is basic and it becomes acidic at the second equivalence point.

In this experiment an accurately weighed sample of solid sodium carbonate is dissolved in a known volume of distilled water and titrated using approximately 0.1 *mol dm*$^{-3}$ HCl solution. Phenolphthalein and then methyl orange indicators will be added close to the equivalence points to verify that the end points are located visually. Phenolphthalein, changes its colour from pink to colourless at pH 9.8. Beyond the first equivalence point phenolphthalein is colourless as the medium is acidic and therefore does not interfere with the subsequently added methyl orange, which changes its colour from orange to red at pH 3.8. The pH at the second equivalence point lies within this colour transition interval. At the second equivalence point, it is necessary to warm the mixture to avoid the effect of CO_2.

Equipment and chemicals required:

Equipment	Chemicals
250 cm^3 Volumetric flask Pipette Burette	0.20 to 0.22 g sample of Na_2CO_3 Phenolphthalein 0.100 mol dm^{-3} HCl solution

Procedure:

- Weigh accurately (to within 0.1 mg) a 0.20 to 0.22 g sample of Na_2CO_3 into a plastic weighing dish. Transfer the solid to a 250 cm^3 volumetric flask.
- Pipet 50.00 cm^3 of distilled water into the volumetric flask. Use the water from the pipette to carefully rinse any solid remaining in the weighing dish into the flask and stir to dissolve the compound. and make up the solution up to the mark by using distilled water.
- Pipette out 50.cm^3 from the above solution to the titration flask and add 4 - 5 drops of phenolphthalein
- Rinse a 50 cm^3 burette with a few cm^3 of approximately 0.100 mol dm^{-3} HCl solution. Fill the burette with the 0.100 mol dm^{-3} HCl solution. Make sure that no air bubbles are in the burette tip.
- Place the titration flask with the Na_2CO_3 solution under the tip of the burette and add HCl solution slowly to the titration flask.
- Stop the addition of acid at the point the colour of the solution become colourless and record the reading of the burette.
- After observing the colour change, add few drops of methyl orange indicator to the solution and continue the addition of HCl.
- Stop the addition of acid at the point at which the colour of the solution becomes red (orange to red) and record the reading of the burette.

- Repeat the titration at least three times and average the volumes at each end point and tabulate data in the following table.

Trial	Volume of 0.100 mol dm^{-3} HCl required for the first end point/ cm^3	Volume of 0.100 mol dm^{-3} HCl required for the second end point/ cm^3
1		
2		
3		
Average		

Post lab questions

1. Two 50 cm^3 samples of 0.2 mol dm^{-3} Na_2CO_3 solution are taken to two separate flasks and add methyl orange to one flask and phenolpthalein to the other. These solutions are titrated with a 0.2 mol dm^{-3} HCl solution. Calculate the two burette readings.

2. 25 cm^3 of a solution containing a mixture of NaOH and Na_2CO_3 was titrated with 0.2 mol dm^{-3} HCl, first with phenolpthalin and then with methyl orange. The burette reading in the presence of phenolpthalien was 25 cm^3. When methyl orange was used the reading was 30 cm^3. Calculate the ratio between [OH^-(aq)] and [Na_2CO_3(aq)].

Experiment 37: Experimental determination of the solubility product of $Ca(OH)_2$

Aims:

1. To determine the solubility product of $Ca(OH)_2$ experimentally
2. To investigate the effects of common-ion, temperature and pH on the solubility of $Ca(OH)_2$

Pre lab questions:

What does a "saturated solution of calcium hydroxide" mean? Explain your answer by providing the equilibrium equation.

Introduction:

Calcium hydroxide is an ionic compound and sparingly soluble in water at a given temperature resulting in the following equilibrium:

$$Ca(OH)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2OH^-(aq)$$

The equilibrium constant for the reaction is the solubility product constant, K*sp*, given by the following.

$$\boldsymbol{K}sp = [Ca^{2+}(aq)][OH^-(aq)]^2$$

This equilibrium is affected by many factors. For example, if $[Ca^{2+}(aq)]$ is increased according to Le'chaterlier principle the equilibrium will shift to the left to keep the value of equilibrium i. e. *Ksp* constant at constant temperature. This increase of $[Ca^{2+}(aq)]$ can be achieved by adding soluble salt like $CaCl_2$ to the initial equilibrium or by dissolving $Ca(OH)_2$) in a $CaCl_2$ solution with known concentration. Similarly, $[OH^-(aq)]$ can also be increased by adding known concentration of NaOH or by dissolving $Ca(OH)_2(s)$ in the solution of NaOH with known concentration. Because of this shift more $Ca(OH)_2(s)$ is produced and hence the solubility of $Ca(OH)_2(s)$ is decreased. This is called the ***"common-ion effect"*** because an ion that is already part of the equilibrium (common to the equilibrium) is added.

Likewise, according to Le'Chaterlier's principle, the opposite is also true. If we can cause the equilibrium to shift to the right then the rate of dissolution is increased more than the rate of crystallization and more solid $Ca(OH)_2(s)$ will dissolve and its solubility increases. This can be achieved by the addition of some reagent which will consume either $Ca^{2+}(aq)$ or $OH^-(aq)$ ions. An easy way for this is the addition of an acid ($H^+(aq)$) ions to the system at a constant temperature. This behaviour of the system helps us to understand the pH effect on the solubility. In the present case, by dissolving $Ca(OH)_2(s)$ in different HNO_3 solutions with known concentrations and calculating the solubility of $Ca(OH)_2(s)$, pH effect can be investigated.

Further, by dissolving $Ca(OH)_2(s)$ in distilled water at different temperatures will enable us to investigate the temperature dependence of the equilibrium constant *Ksp*.

In this experiment, the effect of the factors described above on the solubility of $Ca(OH)_2(s)$ will be investigated experimentally in the laboratory.

Equipment and chemicals required:

Equipment	Chemicals
250 cm^3 titration flask Pipet Burette Beakers Measuring cylinders Thermometer	Solid $Ca(OH)_2$ 0.100 mol dm^{-3} HCl solution 0.100, 0.01 and 1.0 mol dm^{-3} HNO_3 solutions 0.050 and 0.025 mol dm^{-3} NaOH solutions 0.050 and 0.025 mol dm^{-3} $CaCl_2$ solutions Phenolphthalein indicator Distilled water

Procedure

- Prepare saturated stock solution of $Ca(OH)_2$ as follows:

Stock solutions can be prepared by dissolving about 1 g of $Ca(OH)_2(s)$ in 100 cm^3 of each of the following liquids. Note: After dissolving, some solid $Ca(OH)_2(s)$ should remain in the bottom of the beaker.

Stock solution	Saturated Ca(OH)2 solution
1	Distilled water at room temperature (25^{0}C)
2	Distilled water at room temperature (~50^{0}C)
3	Distilled water at room temperature (~90^{0}C)
4	0.050 mol dm^{-3} $CaCl_2$ solution
5	0.025 mol dm^{-3} $CaCl_2$ solution
6	0.050 mol dm^{-3} NaOH solution
7	0.025 mol dm^{-3} NaOH solution
8	1.000 mol dm^{-3} HNO_3 solution
9	1.001 mol dm^{-3} HNO_3 solution
10	0.10 mol dm-3 HNO_3 solution

- Pipette out 25.00 cm^3 of each of the above solutions separately into a titration flask, add a few drops of phenolphthalein indicator and titrate against the 0.100 mol dm^{-3} HCl solution.
- Repeat each titration for another two aliquots of the samples.

Calculations:

1. Calculate [OH^-(aq)] concentration in each case *(use the average value of the burette reading).*
2. First, estimate the value of *Ksp* for the stock solution 1: distilled water at room temperature.
3. Calculate the values of *Ksp* in the other solutions.
4. Compare the values obtained in 3 with that obtained in 2 and comment on your observations.

Post lab questions

1. Explain the variation in concentration of Ca^{2+}(aq) in the saturated $Ca(OH)_2$ solutions as the concentration of OH^-(aq) increases.
2. Discuss the application of *Ksp* in cation group analysis.
3. 25 cm^3 of a saturated $Ca(OH)_2$ solution is titrated with 0.1 mol dm^{-3} HCl and the burette reading is 12.5 cm^3. Calculate the *Ksp* of $Ca(OH)_2$.
4. 25 cm^3 of 0.1 mol dm^{-3} NaOH is mixed with 75 cm^3 of H_2O and then the solution is saturated with $Ca(OH)_2$. 25 cm^3 of that solution is titrated with 0.1 mol dm^{-3} HCl and the obtained burette reading is 14.3 cm^3. Calculate the *Ksp* of $Ca(OH)_2$.

Experiment 38: Experimental determination of the distribution coefficient of ethanoic acid between water and 2 - butanol

Aim:

To examine the dissolution and distribution of a solute in two immiscible liquids

Pre lab questions

1. What does the " distribution coefficient" mean? Explain your answer by providing the equilibrium equation.

Introduction

As we know when two immiscible solvents **A** and **B** are placed in a beaker, they will not mix and instead form separate layers. At a constant temperature, when a solute **X** is added which is soluble in both solvents remains in the same molecular–form in both and does not react with either of them and the system is shaken vigorously, the solute X gets dissolved in both solvents depending on its solubility in each solvent. This nature is mainly used in solvent extraction where a crude (impure) product is obtained in an experiment. For example, the reaction would have been carried out in an aqueous solution and at the end of the reaction there may be unreacted starting materials and unwanted side-products. The aqueous solution is shaken with another immiscible solvent that does not mix with water. The immiscible solvent can be chosen such that the substances that are wanted will dissolve in it, leaving the other substance in the water layer. This procedure can be repeated by adding more of the organic solvent to the aqueous layer, shaking the solutions together, separating them and running of the solution containing the required product. In the cases when a given amount of a solute dissolves between two immiscible liquids at a given temperature, it is necessary to determine the amounts/ concentrations of the solute in both the solvents. Such a situation is described by the partition law in which the ratio of concentrations of the solute is expressed as the equilibrium constant or the partition coefficient, K_D.

This experiment illustrates the distribution of ethanoic acid between two solvents that may be considered to be immiscible, but-1-ol and water. Solutions of the acid in the two solvents are shaken together, allowed to separate and analyzed by titrating a sample of each layer with sodium hydroxide solution.

The procedure is repeated several times, using different concentration of acid and you will then be able to see whether there is any simple relationship between the concentrations of the acid in each solvent.

Equipment and chemicals required:

Equipment	Chemicals
Pipette (10/25 cm^3) Burette Separating funnels Reagent bottles (250 cm^3) Titration flasks (250 ml) Beakers Measuring cylinder (10/25ml)	But-1-ol 150 cm^3 2 mol dm^{-3} ethanoic acid 150 cm^3 ~ 0.50 mol dm^{-3} NaOH 150 cm^3 Phenolphthalein indicator

Procedure

1. Prepare the following mixtures by measuring required volumes of ethanoic acid (CH_3COOH), butanol and distilled water into a separating funnel/ reagent bottle.

Mixture	Butan-1-ol / cm^3	Ethanoic acid(CH_3COOH) / cm^3	Water / cm^3	Volume of 0.50 mol dm^{-3} NaOH / cm^3	
				Organic layer	Aqueous layer
1	20	10	25		
2	20	15	20		
3	20	20	15		
4	20	25	10		
4	20	30	5		

2. Shake the mixture(s) vigorously for about 5 minutes to allow ethanoic acid to dissolve and distribute in both layers. After shaking, rest the separating funnel/ reagent bottle on a experimental bench (table) and allow the layers to separate. At this stage, the top layer is but-1-ol and the bottom layer is water.
3. Using the pipette (use a pipette filler) transfer 10 cm^3 of the upper layer (but-1-ol) into a titration flask. Using a measuring cylinder, add about 25 cm^3 of water to the flask, followed by 3 drops of phenolphthalein indicator. Titrate the resulting mixture against the 0.50 mol dm^{-3} NaOH solution.
4. Using a second pipette, withdraw 10 cm^3 of the aqueous layer from the bottom layer (aqueous) through the top layer and transfer into a titration flask, add 25 cm^3 of distilled water and titrate it against the 0.50 mol dm^{-3} NaOH solution as before.
5. Repeat steps 3 and 4 above with other mixtures prepared and record the results in the corresponding columns in the table.
   NOTE: Steps 3 and 4 can be done as follows if the pipette fillers are not available in the laboratory). After allowing the separation of layers in step 2 just after vigorous shaking of the mixture in a reagent bottle, transfer the mixture into a burette and allow the layers to separate. Now from this you may withdraw the

required volume of water layer (which is in the bottom) first. After the completion of titration of that aqueous layer, you may remove remaining water layer and then the samples from the organic layer can be collected and titrated.

Calculate the concentrations of ethanoic acid in each of the respective titrations and record the results in the table below. Estimate the ratio of

$[CH_3\ COOH]butanol)$
$[CH_3COOH]\ water$

Mixture	$[CH_3COOH]_{butanol}$	$[CH_3COOH]_{water}$	$\frac{[CH_3\ COOH]_{butanol)}}{[CH_3\ COOH]_{water\ )}}$
1			
2			
3			
4			

Comment on the values obtained for $\frac{[CH_3\ COOH]_{butanol)}}{[CH_3COOH]_{water}}$) and hence give the value for the distribution coefficient K_D for the ethanoic acid in butanol and water.

Post lab questions:

1. Why the immiscible solution has to be separated before the ethanoic acid is titrated?

Experiment 39: Experimental determination of the relative positions of commonly available metals in the electrochemical series.

Aims:

1. To observe displacement reactions among metals and metal ions
2. To arrange metals in a series based on the increasing order of their reactivity
3. To determine the relative position of metals in the electrochemical series

Pre lab questions:

1. Write the oxidation and reduction half-reactions for the reactions between
   (a) Mg(s) and a solution of $Al_2(SO_4)_3$(aq) produce Al(s) and a solution of $MgSO_4$(aq).
   (b) Al(s) and a solution of $MgSO_4$(aq) does not produce Mg(s) and a solution of $Al_2(SO_4)_3$(aq)
2. Magnesium is more reactive than aluminum. Predict which of the following combinations of metal and metal ion solution will show evidence to support the above statement
   Mg(s) and a solution of $Al_2(SO_4)_3$(aq) or Al(s) and a solution of $MgSO_4$(aq).

Introduction:

The activity series is a ranking system of metal based on the ease with which they are oxidized. The activity series can be used to predict the reaction between two metal and another metal ion. A metal with higher reactivity oxidizes to reduce a metal ion with lower reactivity. The relative activity of metals can be determined using the reactivity with water, acids and metals ion solutions.

Precautions: In case of contact with skin, chemicals should be washed off with large amounts of water.

Equipment and chemicals required:

Equipment	Chemicals
24 test tubes small pieces of copper, lead, magnesium, zinc and iron	1.0 mol dm^{-3} zinc sulfate solution 30 ml 1.0 mol dm^{-3} ferrous sulfate solution 30 ml 1.0 mol dm^{-3} copper sulfate solution 30 ml 1.0 mol dm-^{3} lead nitrate solution 30 ml 1.0 mol dm^{-3} magnesium sulfate solution 30 ml 1.0 mol dm^{-3} sodium sulfate solution 30 ml

Procedure:

- Clean all test tubes well with distilled water. Label them and place a small piece of metal in the test tube as shown in the table given below. Into each test tube, add 5 ml of each metal ion solution as given in the table below

A-01 tube Cu & Zn^{2+}	**A-02 tube** Cu & Fe^{2+}	**A-03 tube** Cu & Cu^{2+}	**A-04 tube** Cu & Pb^{2+}	**A-05 tube** Cu & Mg^{2+}	**A-06 tube** Cu & Na^{+}
B-01 tube Pb & Zn^{2+}	**B-02 tube** Pb & Fe^{2+}	**B-03 tube** Pb & Cu^{2+}	**B-04 tube** Pb & Pb^{2+}	**B-05 tube** Pb & Mg^{2+}	**B-06 tube** Pb & Na^{+}
C-01 tube Mg & Zn^{2+}	**C-02 tube** Mg & Fe^{2+}	**C-03 tube** Mg & Cu^{2+}	**C-04 tube** Mg & Pb^{2+}	**C-05 tube** Mg & Mg^{2+}	**C-06 tube** Mg &Na^{+}
D-01 tube Fe & Zn^{2+}	**D-02 tube** Fe & Fe^{2+}	**D-03 tube** Fe & Cu^{2+}	**D-04 tube** Fe & Pb^{2+}	**D-05 tube** Fe & Mg^{2+}	**D-06 tube** Fe & Na^{+}

- Observe for a period of 5 – 10 minutes and record any evidence of chemical reactions in each test tube.
- Record the observation in the table given. If no reaction is observed in any test tube record as "no reaction" (NR).

Results:

	$ZnSO_4$	$FeSO_4$	$CuSO_4$	$Pb(NO_3)_2$	$MgSO_4$	Na_2SO_4
Cu						
Pb						
Mg						
Fe						
Zn						

Discussion:

a. Which metal reacts with most of the metal ion solutions?
b. Which metal reacts with fewest of the metal ion solutions?
c. Compare the general trend in the reactivity of Cu, Fe, Mg, Pb and Zn and rank the metals from the most active (first) to the least active (last).

Post lab questions:

1. Write the balanced chemical equations for the reactions of the most active metal tested in this experiment.

Experiment 40 : Preparation of standard Ag(s), AgCl(s)| Cl^-(aq) electrode.

Aim:

To understand the preparation method of standard Ag(s), AgCl(s)| Cl^-(aq) electrode.

Pre lab questions:

1. What is a standard electrode?
2. Write anode, cathode and the balanced cell reactions for an electrochemical cell consist of a standard hydrogen electrode connected to a Zn/Zn^{2+} ion electrode.
3. Write the full electrochemical cell notation for the electrochemical cell given in part (2).
4. What are the standard electrode potentials of standard Ag(s), AgCl(s) | Cl^-(aq) and calomel electrodes with respect to standard hydrogen electrode?

Introduction:

Standard electrodes are also known as reference electrodes. Standard electrodes have their own defined and stable potential. Standard hydrogen electrode (SHE) is assigned an arbitrary potential value of zero. SHE is the most accepted reference electrode globally. Reference electrodes can be divided into two groups.

1. Primary reference electrode: Standard hydrogen electrode
2. Secondary reference electrode: Reference electrodes other than standard hydrogen electrode. For example, silver-silver chloride and saturated calomel electrodes. Secondary reference electrodes are commonly used due to the ease of handling and preparation.

Precautions:

Concentrated HNO_3 is extremely corrosive. Students must wear eye protection while handling conc HNO_3.

Equipment	Chemicals
Variable DC potential supplier Silver wire (diameter 1 mm) Soldering iron Empty transparent plastic pen outer casing Circuit wire (1 m) Insulating adhesive tape Graphite electrode (use a both end sharpen pencil)	Saturated KCl solution Conc. HNO_3 Silicone sealer

Procedure:

- Dissect 5 cm wire piece of silver wire (silver wire can get from a local goldsmith) and sand the wire well using a fine sandpaper. Solder one end of the silver wire with a small piece (30 cm) of circuit wire. Wash the unsoldered end of the silver wire with conc. HNO_3. Then wash the wire with distilled water.
- Use 100 cm^3 beaker and add 20 cm^3 of 3 mol dm^{-3} KCl and 40 cm^3 of deionized water to prepare a dilute KCl solution. Immerse the unsoldered end of the silver wire (3 cm) and both ends sharpen pencil in this solution. Connect the positive terminal of the variable DC voltage supply to the circuit wire connected to the silver wire and the other terminal with both ends sharpen pencil.
- Increase the applied voltage till bubbles start to appear, and leave the setup connected to the power supply for few minutes. With the time the immersed section of the silver wire become brown in colour due to the production of AgCl. Disconnect the power supply when AgCl is fully developed around silver wire.

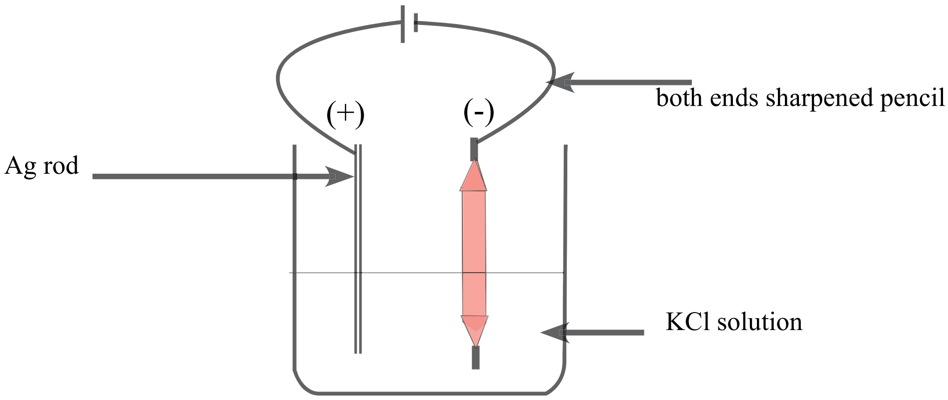


Figure 41.1: Preparing AgCl coating on the silver rod

- Separate a 7 cm size section from the pen casing and wash with deionized water.
- Mix agar gel and 3 mol dm^{-3} KCl; heat well. It is enough to fill up to about 2 cm height. Put the empty pen casing in a boiling tube, horizontally and then cool for 24 hours.

Note:

1. Instead of empty pen casing, medical syringe / auto pipette tip can be used.
2. AgCl(s) is precipitated on Ag wire by dipping the cleaned Ag wire for 1-2 min in a solution of bleaching powder.

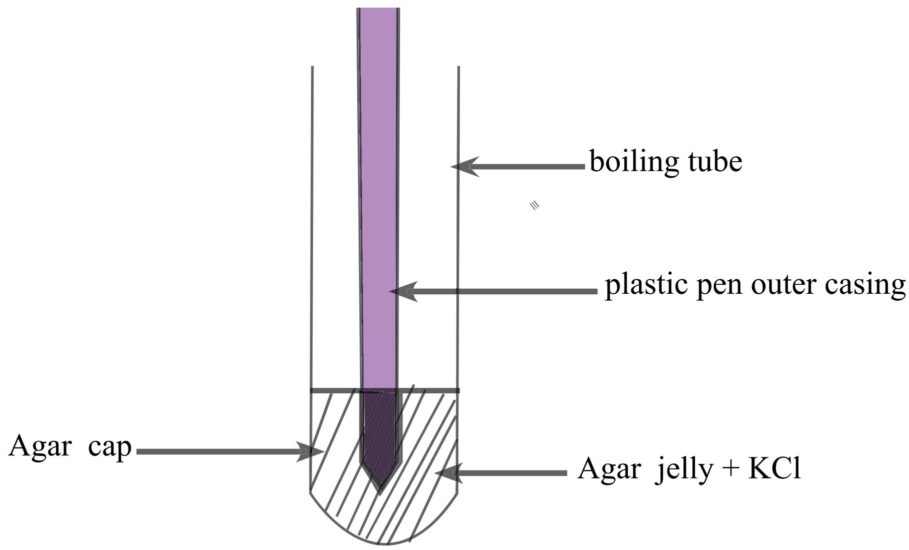


Figure 41.1: Preparation of agar plug

- Fill the plastic pen casing with saturated KCl solution using a medical syringe. Insert previously prepared silver wire to the empty plastic pen case that one end was sealed with agar.
- Then, seal the upper end of the pen case using adhesive tapes and silicone sealer as shown in the diagram.

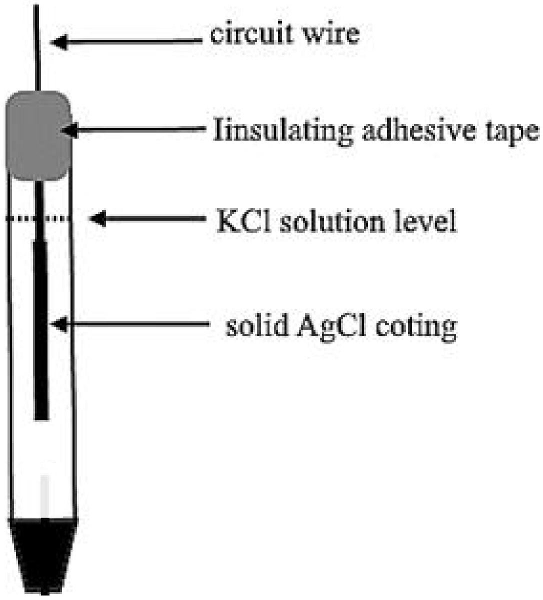


Figure 41.2: Finished Ag(s) /AgCl(s) /Cl- electrode

Post Lab questions:

1) Discuss the limitation of developed Ag/AgCl electrode
   (Hint: the silver wire exposed can be reacted under harsh conditions)
2) Measure the potentials of metal/metal ion electrodes using the developed Ag/AgCl electrode.
3) Discuss the importance of keeping the inner filling KCl solution concentration unchanged.
4) What can happen to the electrode potential when a 3 mol dm^{-3} KCl solution is used instead of saturated KCl solution to construct the Ag/AgCl electrode?
5) Write anode, cathode and the balance full reactions for an electrochemical cell consist of a standard Ag/AgCl electrode connected to a Zn/zinc ion electrode.
6) Write the full electrochemical cell notation for the electrochemical cell given in part (2).

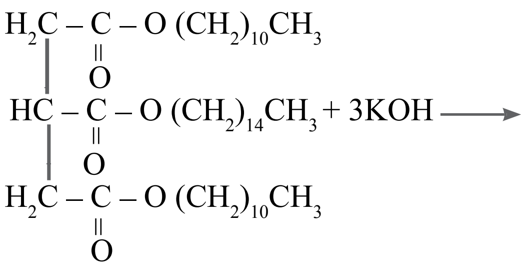
Experiment 41: Preparation of soap sample in the laboratory

Aim:

To learn the skills required for the soap production under laboratory conditions

Pre lab questions:

1. Complete and balance the following reaction that occurs in the soap production process.



2. List the main constituents in coconut oil and chemical structure of these constituents.
3. Write the reaction of hydrolysis of the ester.
4. Discuss the differences between plant oils and animal fatty acids.

   Describe how detergents differ structurally from soap.

Introduction:

Soap making or saponification process dates back to 600 BC. Soap production process is based on fatty acids such as lauric or palmitic fatty acids that can be found in plant oils. Fat and vegetable oils are triglycerides. Saponification is the hydrolysis of an ester to produce a carboxylic acid salt and an alcohol using a base as the catalyst. For the soap production, both NaOH and KOH can be used. Because of that soaps can be generally called sodium or potassium salts of long chain fatty acids. When soap is dissolved in water, hydrolysis occurs to some extent to produce long chain carboxylic acids.

In the presence of acids or Ca^{2+} and Mg^{2+} ions, water-soluble soap molecules are converted into water-insoluble forms. This can reduce the cleaning action of the soap.

Precautions:

Concentrated sodium hydroxide is extremely caustic and corrosive. Students must wear eye protection while performing the experiment.

Equipment and chemicals required:

Equipment	Chemicals
Bunsen burner Buchner funnel Beakers Magnetic stirer	Coconut oil NaOH NaCl

Procedure:

- Use a 250 cm^3 conical flask and add 25 cm^3 of vegetable oil or coconut oil, 20 cm^3 of ethanol and 25 cm^3 of 6 mol dm^{-3} NaOH into the flask. Stir the mixture with a stirring rod.
- Heat the mixture using a boiling water bath and stir continually to prevent the formation of form as shown in the Figure below.

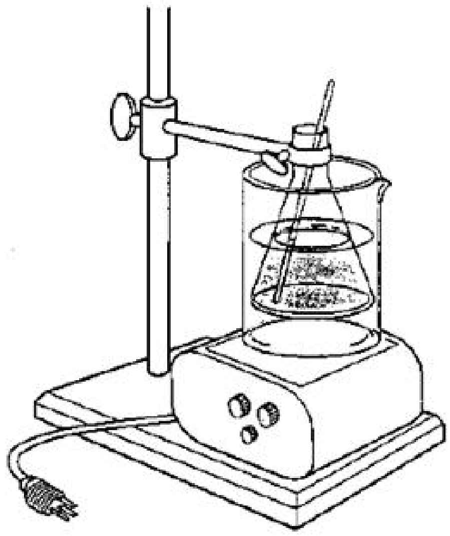


Figure 42.1 Set-up used for soap production

- Heat the mixture for a period of 10 - 15 minutes. The mixture will transform to a paste at the end of heating.
- After the heating, remove the mixture (now soap) from the boiling water bath and cool down using an ice-bath.
- Add 150 cm^3 of saturated NaCl to the cooled soap mixture and filter the soap using a Buchner funnel with a weighted filter paper. A sketch of the filter set-up is shown in the Figure given below (alternately gravity filtration can also be used.)

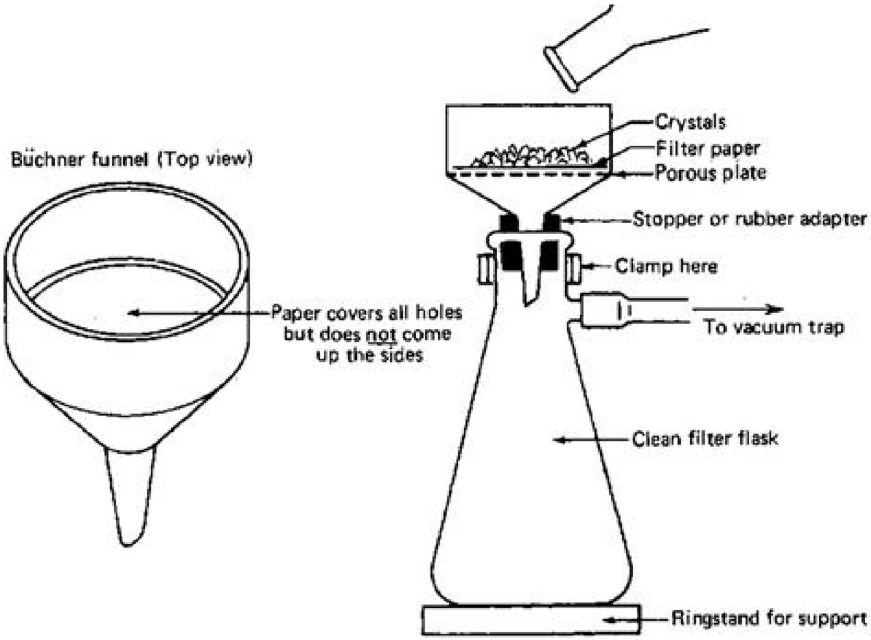


Figure 42.2 Filtrations set-up used for soap separation

- Remove the collected soap with the filter paper and press with paper towels to dry it.

Calculations:

1. Assume that the coconut oil contains 80% lauric acid and 20% none soap producing compounds. Taking molar volume of lauric acid as 221.3 cm^3 mol 1. calculate the theoretical yield when 25 cm^3 of this coconut oil is used.

2. Calculate the efficiency of the soap manufacturing process if the same coconut oil is used.

Discussion:

a. Explain the action of ethanol in this production process.
b. Discuss possible environmental effects of soap production process and explain how you can suggest methods to make the process more environment friendly.
c. Discuss how detergents can produce the cleaning action in the presence of calcium and magnesium salts (hard water) .
d. Explain why detergents can produce a greater impact on the environment than soap.

Post lab questions:

1. What are the possible intermolecular interactions of a soap molecule that can be formed with water and dirt molecules?

2. Soap with higher pH is not recommended for human use. Investigate the industrial process to eliminate excess unreacted NaOH in soap.

3. Calculate the weight of the precipitate formed when 1 g of soap (100% sodium laurate) reacts with excess $CaCl_2$.

4. What is the main by-product formed in the soap manufacturing process and explain another industrial process that can use this by-product as a starting material?

Experiment 42: Extraction of essential oils by steam distillation [Extracting cinnamon oil from cinnamon leaves by steam distillation]

Aim:

To develop the skills required for the extraction of essential oils from selected natural sources

Pre lab questions:

1. What are the major components of given essential oils in the table? Complete the table.

Oil	Major component	Use
Cinnamon oil		
Clove oil		
Oil from orange peel		
Lemon grass oil		

2. Explain how steam distillation is used to isolate essential oils.

3. Why steam distillation is preferred over regular distillation for extracting essential oils?
4. Give two other methods that can be used to extract essential oils.

Introduction:

Steam distillation is one of the methods used for obtaining essential oils from various plant based substances. Steam distillation is used to isolate volatile substances that have high boiling points. When two immiscible liquids are distilled together, the amounts of each component in the distillate are constant. Also, the boiling point of the mixture is lower than that of either of the individual components. This process allows high-boiling compounds, which can decompose before they boil if heated alone, to be distilled at temperatures below 100 °C.

So, the boiling of the water + oil mixture occurs when sum of the pure vapour pressures of oil (P_{oil}) and water (P_{water}) is equal to the atmospheric pressure. Since $P°_{water} >> P°oil$, the mixture will boil at a temperature slightly less than the normal boiling point of water. This assure the evaporation of the essential oil under mild conditions. The molar fraction of the final distillate can be given using the equation below.

$$\frac{n_{(oil)}}{n_{(water)}} = \frac{P_{(oil)}}{P_{(water)}}$$

Precautions: Students must wear eye protection while performing the experiment.

Equipment and chemicals required:

Equipment	Chemicals
Bunsen burner Molar and pestle Round bottom flask or distillation flask Beakers Distillation apparatus with a thermometer Condenser Separating funnel Heating mantle Steam bath Glass rod Measuring cylinder Erlenmeyer flask	Selected spice (Cinnamon / Clove/ Lemon or Orange peel) about 5 g Drying agent

Procedure:

Week 01

- Weigh 5.0 g of selected spice (cinnamon or clove) or lemon/ orange peels and grind it or cut into small pieces with a mortar and a pestle. Transfer the sample into a 250 cm^3 round-bottomed flask (distillation flask). Add 50 cm^3 of distilled water into the round-bottomed flask with the sample.
- Set up the distillation apparatus as shown in the Figure. A beaker or a second round-bottomed flask can be used as the receiving flask.
- Turn on the condenser water to a slow flow rate, and heat the flask with direct flame or the heating mantle so that the liquid boils gently. Record the temperature when the first drop comes over. Try to obtain a steady rate of distillation, around 1 drop per 2-3 seconds. Continue distilling until you have collected about 30 mL of distillate. Record the temperature again at the end of collection.
- Remove the flask from the heat and turn off the heating mantle.

Week 02

- Place all of the distillate into a separatory funnel. Add 10 cm^3 of dichloromethane/ diethyl ether, shake, and allow the layers to separate. Collect the organic layer into a clean Erlenmeyer flask. Repeat this extraction step two more times and collect all three organic fractions into a beaker.
- Add a few small scoops of drying agent (anhydrous sodium sulfate); add until the drying agent no longer clumps together when swirled (should look like a snow-globe). Allow to stand for 10-15 minutes, stirring occasionally.
- Decant the liquid into a small pre-weighed round bottom flask and arrange a

distillation set-up in order to separate dichloromethane / diethyl ether. Heat the round bottom flask having organic layer using a steam bath and collect the solvent (dichloromethane / diethyl ether). Continue the distillation until the receiving of solvent is over. (Don't use Bunsen burner or flames, use heating mantle.)

- Dismantle the distillation setup and dry outside (water droplets) of the round bottom flask. Weigh round bottom flask having essential oil. Place the round bottom flask on steam bath for few minutes (10 minutes) in order to boil off solvent residue. Weigh it again and compare the weight loss. (Don't use Bunsen burner or flames, use heating mantle.)

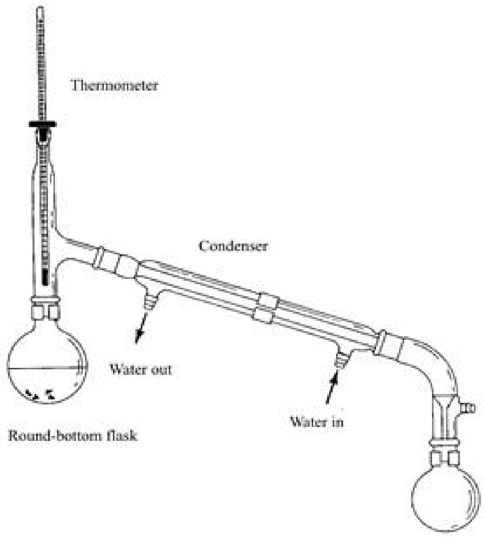


Figure 43.1 Set-up used for the steam distillation

Discussion:

a. Discuss the criteria you would use to decide whether distillation is an appropriate method to extract a natural product from a plant based source.
b. Discuss suitable methods to make this process more environment (green) friendly.

Post lab questions:

1. Why does the distillate that you collect initially appear cloudy?
2. What is a drying agent and how does it work?
3. Suppose you are going to extract an essential oil with the vapour pressure at 100 °C is known to be half that of the essential oil extracted in this experiment. What consequence would this have on the amount of distillate required per mole of the natural product?

Experiment 43: Preparation of biodiesel.

Aims:

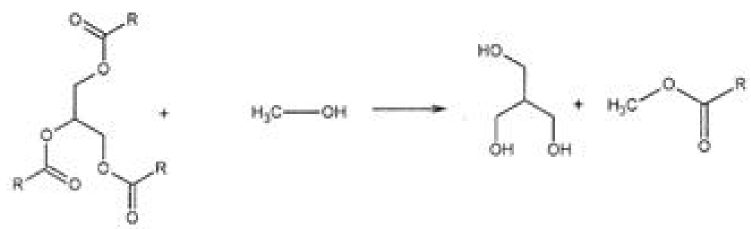
1. To prepare an environmental friendly liquid fuel source
2. To understand the advantages of biodiesel

Pre lab questions:

1. List the advantages and disadvantages of biodiesel

Introduction:

Synthesis of biodiesel is a process catalyzed by NaOH. In the first step, NaOH reacts with methanol to produce methoxide anion. In the second step, methoxide nucleophile attacks a carbonyl carbon of vegetable oil to produce methyl esters. NaOH is reproduced at the end of the process. Glycerol is produced as the main byproduct in the biodiesel production reaction. Glycerol can be used as an ingredient for many other industrial process. Plant oil used in various food industries can be recycled to produce biodiesel.



Precautions:

Students must wear eye protection while performing the experiment.

Equipment and chemicals required

Equipment	Chemicals
Erlenmeyer flask (250 cm^{-3}) Beaker (250 cm^{-3}) Separatory funnel (250 cm^{-3})	Vegetable oil (used or unused) NaOH methanol

Procedure:

- Add 0.5g of NaOH into a 250 cm^3 Erlenmeyer flask with methanol and stir vigorously unitl all of the NaOH is completely dissolved.
- Warm up 100 cm^{-3} of unused vegetable oil to about 40^0C in 250 cm^{-3} beaker. Used vegetable oil must be filtered and remove water before using for the biodiesel production process.
- Introduce the vegetable oil sample into the NaOH-methanol mixture while stirring. Continue stirring until two layers appear.
- Transfer the content to a separatory funnel and take out the top layer. The bottom layer is glycerol and the top layer is methyl ester.
- Soak a half of a paper strip, burn it and observer the rate of combustion.

Results and observation

Write the observations in steps 3 and 4.

Discussion:

- Discuss the importance of using biodiesel and other renewable liquid fuels.
- Discuss how various countries use renewable liquid fuels in motor vehicles.

Post lab questions:

1. In this reaction, 30 cm^3 of oil (molar mass = 900 g/mol and density = 0.92g/cm^3) and 8 cm^3 of methanol (density = 0.79 g/cm^3) are used. What is the limiting reagent of this reaction ?
2. List industries that use glycerin as a starting material.

Experiment 44: Determination of the percentage of aacetic acid in Vineger.

Aim:

To determine the molarity and the percent by mass of acetic acid in vinegar by titrating with sodium hydroxide

Pre lab questions:

1. Write the complete balanced equation for the reaction between CH_3COOH and NaOH. Label the acid, conjugate acid, base and conjugate base for this reaction.
2. What is an acid-base indicator and how does it help to perform an accurate titration?
3. Explain the difference between end-point and equivalence point of a titration.
4. Name an indicator suitable for the titration between CH_3COOH and NaOH

Introduction:

Acid-base titration is a method used to determine the amount of acid or base in a given sample. In this experiment, the amount of acetic acid in vinegar is determined by adding a solution of NaOH with a known concentration until all acetic acid in the sample is fully neutralized. An acid-base indicator is used to indicate the point where the acid is fully neutralized. To determine the concertation of acetic acid, the volume of the NaOH is added into the sample must be known.

At the equivalence point all acetic acid is neutralized by NaOH.

Amount of moles of NaOH added = Amount of moles of acetic acid initially in the volume used for the titration

$$\text{mass of acetic acid} = \text{mole of acetic acid} \times M_{\text{acetic acid}}$$
$$= \text{mole of NaOH} \times M_{\text{acetic acid}}$$
$$= C_{NaOH} \times \frac{V_{NaOH}}{1000} \times M_{\text{acetic acid}}$$

Precautions: Students must wear eye protection while performing the experiment.

Equipment and chemicals required:

Equipment	Chemicals
Burette Titration flasks Pipet Small funnel Beakers	0.2 mol dm^{-3} standardized NaOH Phenolphthalein indicator Vinegar

Procedure:

- Rinse a 25 cm^3 burette (or 50 cm^3 burette) first with distilled water and with about 5 cm^3 of 0.2 mol dm^{-3} NaOH solution. After rinsing, using a clean and dry funnel, fill the burette with 0.2 mol dm-^{3} NaOH solution to about 2 cm^3 above the 0.00 cm^3 mark. Slowly turn the stopcock to allow the solution to drain till the solution level drops to 0.00 cm^3 mark. The air bubbles must be completely removed from the solution.
- Using a 5.00 cm^3 volumetric pipette, add 5.00 m^3 vinegar to a 100 cm^3 titration flask. Allow the vinegar to drain completely from the pipette by holding the pipette in such a manner that its tip touches the wall of the flask. Add a few drops of phenolphthalein indicator to the flask and about 10 cm^3 of distilled water. The distilled water is added to dilute the natural colour of some commercial vinegar.

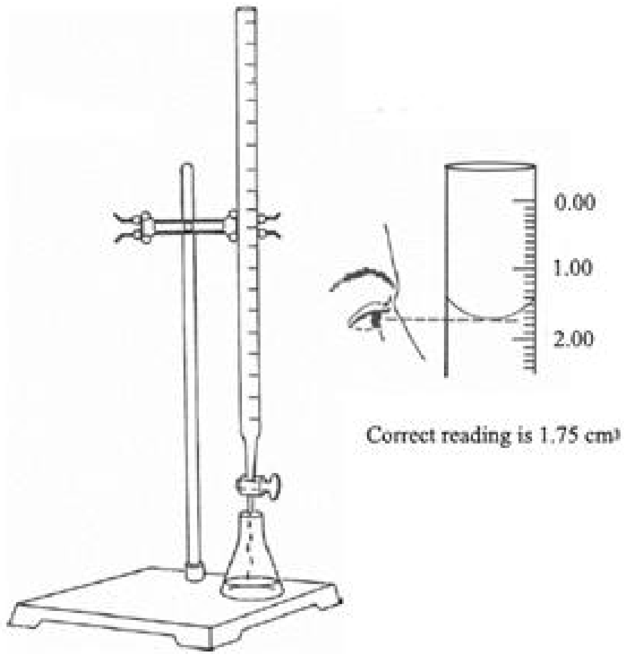


Figure 45.1 Titration set-up used for the titration between vinegar and NaOH

- While holding the neck of the Erlenmeyer flask in your left hand and swirling it, open the stopcock of the burette slightly with your right hand add the base to the flask drop wise. Continue the titration until a faint permanent pink colouration appears.
- Repeat the procedure twice in steps 1–3 with new 5 cm^3 vinegar samples.

Results	Trial 01	Trial 02	Trial 03
Volume of vinegar sample	…………….	…………….	…………….
Molarity of NaOH solution	…………….	…………….	…………….
Initial reading of NaOH in burette	…………….	…………….	…………….
Final reading of NaOH in burette	…………….	…………….	…………….
Volume of NaOH used in titration	…………….	…………….	…………….
Average volume of NaOH used in titration		……………	……………
Molarity of acetic acid in vinegar		……………	……………

Calculations:

1. Calculate the weight percentage (w/v) of acetic acid in vinegar.

2. Calculate the percentage error in the value marked on the vinegar bottle by the manufacturer.

Discussion:

a. Discuss the accuracy of the concentration of the NaOH solution you have prepared for this experiment.
b. Discuss the possible reasons for any deviations between the experimental and marked percentages of vinegar by weight.

Post lab questions:

1. A student carried out the experiment but did not follow the directions exactly as written. What effect would the following actions have on the experiment?
   a. The student took a sample of the vinegar with a 5 cm^3 volumetric pipette and then emptied all the liquid into an Erlenmeyer flask by blowing out the sample.
   b. Air was not excluded from the burette when it was filled with NaOH solution. An initial burette reading was taken; then during the titration, the air bubble came out. When the titration reached the end point, a final reading was taken.
   c. Using methyl orange indicator instead of phenolphthalein.
2. A super market store sells vinegar in 500 cm^3 bottles. Based on your results, how many grams of acetic acid are in a single bottle?
3. Sketch the pH vs. volume of NaOH variation for the titration between vinegar and NaOH.

Experiment 45 : Determination of dissolved oxygen level in water by Winkler method

Aim:

To determine the level of dissolved oxygen in a sample of water using the Winkler method

Pre lab questions:

1. Explain why the dissolved oxygen content of an environmental sample of water is important.
2. Explain what contributes or favours an increased value of dissolved oxygen in water.
3. What are the main types of pollutants that affect the level of dissolved oxygen in water?

Introduction:

Dissolved oxygen (DO) level is one of the most important water quality parameters. Discharge of warm effluents can directly affect the DO levels of water bodies. In addition, the release of organic pollutants or nutrients to water bodies can also reduce the DO levels. DO level of a water body can be used to determine whether the system is anaerobic or aerobic to decide the possibility for the aquatic living organisms to survive.

Precautions: Students must wear eye protection while performing the experiment.

Equipment and chemicals required:

Equipment	Chemicals
Dark colour stoppered bottles Calibrated pipettes (capable of dispensing) 1 cm^3, 10 cm^3, and 50 cm^3 aliquots Titration flasks Beakers	3 mol dm^{-3} $MnCl_2$ 4 mol dm^{-3} KI in 8 mol dm^{-3} NaOH 50% H_2SO_4 Starch indicator 0.18mol dm^{-3} $Na_2S_2O_3$ 0.00167mol dm^{-3} KIO_3 standard solution

Procedure:

Collection of water sample:

- Rinse the sample bottle twice with the sample water before collecting the sample. After rinsing, slowly fill the bottle avoiding bubbles introduced into the water. Then place the stopper of the bottle.
- Immediately after obtaining the water sample, introduce 1 cm^3 of 3 mol dm^{-3} $MnCl_2$ and 1 cm^3 KI (in 8 mol dm^{-3}NaOH). This must be done by dipping the tip of the pipette into the water sample.

Standardization of $Na_2S_2O_3$ solution

- Add 10.00 cm^3 of KIO_3 and 1 cm^3 of H_2SO_4 into an Erlenmeyer flask. Add 1 cm^3 of KI, mix well, cover with a watch glass and store under dark for a period of 5 – 10 min.
- Titrate this mixture with the $Na_2S_2O_3$ solution until a pale yellow colour is obtained. Add a few drops of starch solution, and continue the titration until the solution is colourless.
- Record the volume of $Na_2S_2O_3$ added.

Sample analysis

- Immediately prior to the analysis add 1 cm^3 of 50% H_2SO_4 into the bottle with the water sample. Then invert the sample bottle few times. At this point, the water sample must turn into deep yellow colour.
- Titrate the water sample using the standardized $Na_2S_2O_3$ solution and record the volume of $Na_2S_2O_3$ added using starch indicator.

Results and observation

Sample No: 01
Temperature of the sample:...
Sample volume used for titration ....
Colour of the precipitate formed after the first two reactant additions:......
Exact concentration of the $Na_2S_2O_3$ solution:.....................................

Sample	volume of the titrant cm^3	DO concentration/mgL^{-1}
Tap water		

Discussion:

a. Write the balance chemical equations for the reactions occurred in the DO determination process.

b Explain how DO depends on the temperature of water and the partial pressure of oxygen in the atmosphere.

Post lab questions:

1. Does the DO level of water differ with the time of the sample collection? What may be the causes of such differences?
2. Why is it important to fix the sample on site soon after collection?

Azide - Winkler Method

